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PROSPECTING AND MINING OF COPPER ORE AT SANTA RITA, NEW MEXICO

BY

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Cooperation with the United States Geological Survey

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PROSPECTING AND MINING OF COPPER ORE AT SANTA RITA, N. MEX.

By Donald F. MacDonald and Charles Enzian.

INTRODUCTION.

It is a far cry from the small and uncertain mining efforts of 50 years ago to the splendidly equipped and solidly financed organizations that now handle thousands of tons of ore per day. The old mining was largely speculative; the new is practically on a manufacturing basis. In the old days of mining the aim was, chiefly, to find comparatively rich ore; modern practice looks more toward efficiency in mining and milling, so that vast tonnages of low-grade material may be worked at a profit. The old operators had faith and hoped for good returns; the new ascertain with a fair degree of precision what the available tonnage is, what the cost of mining and treating the ore will be, the time necessary to exhaust the deposit, the capital required, and the net profits to be made. The old mining was financed on a highly speculative basis, and the value of mining stock was largely controlled by the imaginations of the promoters and of the individual holders. In the new régime the financing of mines is often as conservative as the financing of manufactures or railroads, and the value of stock, quoted on the leading exchanges, is controlled by the carefully estimated profits in sight. Of course, with every legitimate industrial enterprise go certain business risks, and mining is not exempted from these. Then, too, valueless mining as well as valueless industrial stocks are sometimes offered for sale by the intentionally dishonest, or the ignorant. However, the prospective purchaser of a mine nowadays, if he is willing to pay for the services of reputable mining engineers, can obtain information on which to base an opinion as to the quality of his investment.

The relatively recent development of cheap and efficient methods of mining and of ore dressing has given value to low-grade ore bodies that a few years ago were almost valueless. The iron mines of northern Minnesota have been fruitful in developing efficiency in mining methods, notably the open-cut methods in which steam shovels strip off the overburden and then load the ore directly into railroad trains for transport to the furnace. In 1907 open-cut mining was applied to the low-grade copper ores of Bingham, Utah, with splendid results, and since then it has won a most important place in the mining of large masses of low-grade ores in several other mining regions, especially at Santa Rita, N. Mex.

Efficiency in mining as in other industries implies attention to detail, and the larger the scale of operations the more important may details become. For this reason the accompanying study of the methods used at Santa Rita will, it is believed, be of help to all engineers who have occasion to handle large masses of material.

ACKNOWLEDGMENTS.

Any merit that this report may have must be largely attributed to the splendid opportunity for study afforded the authors by the Chino Copper Co. To the manager, Mr. John M. Sully, the authors are deeply indebted for kindly interest and every facility to investigate the methods in use at the mines and mill of the company. For many courtesies and much help they are also under obligations to Horace Moses, superintendent; L. E. Foster, assistant superintendent; M. J. McGrath, chief engineer; Harry Thorne, explosives foreman, and many other officers of the Chino company's mine as well as to W. H. Janney, superintendent, and W. T. MacDonald, assistant superintendent, of the Hurley mill. Nearly two months were spent by the authors in field work at Santa Rita in the summer of 1914.

The outline of the geologic conditions that follows is largely from notes furnished by Spencer[a] and by Paige;[b] others[c] have also furnished valuable data bearing on the mining conditions of the region.

LOCATION AND GENERAL GEOGRAPHY OF SANTA RITA DISTRICT.

Santa Rita is on the mountain-bearing plateau of southwestern New Mexico. It is nearly 150 miles northwest of El Paso, Tex., and about 50 miles in the same general direction from Deming, N. Mex. It lies about a dozen miles east of Silver City, N. Mex., and is on Santa Rita Creek, not far from where the latter has its rise in the southeastern foothills of the Black Range. A branch of the Santa Fe Railroad connects Deming, N. Mex., on the Southern Pacific main line, with Silver City. From Whitewater, N. Mex., on this branch line, a subbranch leads to Santa Rita and to Fierro, N. Mex. (See maps, Pls. I and II.)

Santa Rita is in the central mining district of Grant County. In its vicinity the plateau is about 6,300 feet high, but some of the neighboring peaks reach altitudes of nearly 8,000 feet.

The climate is dry, bracing, and sunny. Even in the bright hot summer months the nights are cool. In winter, clear, cool weather, with occasional snow, adds to the stimulating effect of the climate.

---

[a] Spencer, A. C., A detailed report on the geology of Santa Rita, in course of publication by the United States Geological Survey.

[b] Paige, Sidney, Silver City folio (No. 199), Geol. Atlas U. S., U. S. Geol. Survey, 1916, 20 pp.

[c] Lindgren, W., Graton, L. C., and Gordon, C. H., The ore deposits of New Mexico: U. S. Geol. Survey Prof. Paper 68, 1910, 361 pp.

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The following table gives the maximum and minimum temperatures and monthly evaporation for the year 1914. The monthly mean of the daily maximum and minimum temperatures is given under "mean maximum" and "mean minimum."

Data on temperature and precipitation at Santa Rita, N. Mex., during 1914, by months.

Month.	Temperature.[a]				Evaporation.		Precipitation.[b]	
	Maximum.	Minimum.	Mean maximum.	Mean minimum.	During month.	Total.	During month.	Total.
1914.	° *F.*	° *F.*	° *F.*	° *F.*	*Inches.*	*Inches.*	*Inches.*	*Inches.*
January	64	14	52.27	28.15	4.329	4.329	0.58	0.58
February	62	22	51.39	28.95	4.266	8.595	0.43	1.01
March	70	24	57.63	32.66	7.448	16.043	0.66	1.67
April	78	30	66.88	41.18	11.507	27.550	0.02	1.69
May	84	31	74.23	48.27	12.416	39.966	1.00	2.69
June	91	48	81.82	57.50	12.253	52.219	1.51	4.20
July	85	52	78.11	58.76	7.612	59.831	7.51	11.71
August	88	53	80.98	58.66	9.151	68.982	2.98	14.69
September	83	46	76.77	54.12	8.214	77.196	0.75	15.44
October	78	37	65.27	43.03	5.964	83.160	3.58	19.02
November	64	30	57.73	36.48	3.584	86.744	0.93	19.95
December	50	15	42.94	27.15	1.636	88.380	4.70	24.65

[a] Elevation of gaging station, 6,311.5 feet.

[b] Snow reduced to equivalent rainfall.

The streams are not large, and the country is not particularly well watered, either for mining or for agriculture. Marketable timber is scarce except in the higher mountains, and even there it is inclined to be scrubby. The timber used at the Santa Rita mines is brought in by rail, as the available local supply, never great, was exhausted some years ago.

Santa Rita and the neighboring regions offer an attractive field to the prospector; the climate is delightful, the country open and easy to traverse, and the rock outcrops fairly favorable.

GEOLOGIC CONDITIONS GOVERNING MINING IN SANTA RITA DISTRICT.[a]

The ore bodies at Santa Rita are associated with quartz-monzonite and quartz-monzonite porphyry intrusions, which cut limestones, sandstones and shales. These intrusives are of post-Cretaceous date, and are older than tuffs, rhyolites, and andesites in the vicinity, which are regarded as of Miocene age. Of the three types of intrusives near the ore, the oldest and the one constituting the largest mass is a quartz-porphyry; the next in point of age is an essentially even-grained quartz-bearing monzonite, containing idiomorphic feldspars and occurring as a stock of moderate size; the youngest is a porphyritic rock, locally characterized by large crystals of quartz, which occurs in the form of dikes. The sedimentary series cut by these intrusives consists of limestones of Carboniferous (Pe vanian) age, and of Cretaceous shales and sandstones.

---

[a] Notes on the geology of the region were furnished by Dr. A. C. Spencer, whose complete report on the geology of the Santa Rita district will be published by the United States Geological Survey.

The alteration of the rocks in the vicinity of the ore deposit been marked. Within a rudely elliptical area, measuring 7,000 feet east and west and more than 10,000 feet north and s the prevolcanic rocks were thoroughly permeated by metamorph solutions. These solutions have sericitized the shales and charged them with pyrite; they have silicified and pyritized sandstones, and have changed the limestones in many place chlorite-sericite rocks, carrying large amounts of pyrite and lo much magnetite. All the igneous rocks are somewhat minerali as shown by the presence of pyrite. The older quartz-porphyr practically everywhere completely sericitized, but this sort of alt tion is much less general, and is locally lacking in the you intrusives.

In the main, the mineralization is of the type that is produced general metasomatic alteration of the rocks. Highly individuali veins are essentially lacking, though reticulating fractures in rocks are locally healed by quartz fillings. The date of the miner ization is not precisely known, but on the south the altered roc are overlapped by unmetamorphosed volcanics, indicating that t mineralization was essentially complete before the extravasation the latter in Miocene time.

ORE DEPOSITS.

The copper ores of Santa Rita are mainly low-grade deposits i which the copper minerals are disseminated through the rocks, an various rocks, both sedimentary and igneous, are contained in th ore bodies. Metamorphism and primary sulphide deposition resulte from the action of hot solutions of magmatic origin. Almost everywhere the altered rocks carry at least small amounts of copper, but material of ore grade has been formed mainly through the enrichment of relatively lean rock by the downward movement of water.

The principal copper mineral of the sulphide ores is chalcocite, but metallic copper and cuprite are locally present. Chalcopyrite, though seemingly not abundant in the ores, must have been the primary mineral from which most, if not all, of the secondarily deposited copper has been derived. Malachite occurs in much of the weathered capping, and is intermixed in varying proportions with other copper minerals in the upper parts of the sulphide ore bodies.

Rich copper ores that were mined in former years came from a series of ore bodies disposed along a curving belt 400 to 800 feet wide and about 6,000 feet long. This ore belt is comprised in the wider and more extensive zone which includes the principal ore bodies of much lower average grade that have been developed by the Chino

Copper Co. As now outlined (see Pl. III) the ore zone is an approximately elliptical annulus 500 to 1,000 feet wide, with an inside shorter diameter of 2,000 feet and a longer diameter of 3,000 feet. The principal axis of the ellipse trends northwest. The ores are of variable composition, as the different rocks mentioned have entered into the ore bodies, and each rock has been differently affected by the metamorphosing agents.

In the main the results of churn-drill prospecting show that the ore bodies are rather definitely though not sharply limited downward, and this, together with the fact that chalcocite replaces pyrite, proves that the ores owe their present copper content partly to processes of enrichment due to the downward movement of surface waters.

The weathered overburden that caps the sulphide ores varies in thickness from a few feet to 150 feet or more, the average being about 85 feet. In general the ore bodies measure more than 100 feet vertically, but what may be called the bottom surface is extremely irregular. Thus in one section of the ore area, material carrying chalcocite and metallic copper has been found continuing to depths as great as 1,300 feet, and in several places the ore bodies are 400 to 500 feet thick.

The variations in thickness, and the fact that the segregations that are large enough and of sufficiently high grade to constitute ore occur in a rather definite zone, are thought to be due to differences in the conditions of underground drainage.

The variations in the mineral constitution of the ores are wide. Different sorts and grades of ore are rather intricately intermixed, so that maintenance of the output of a mine at any average mineral composition is always difficult and at times impossible. The chief variations of practical moment are, first, in the relative amounts of copper minerals to pyrite and magnetite, and, second, in the proportions of heavy copper minerals to malachite. In the milling of the ores a high iron content—that is, a high proportion of pyrite and magnetite—tends to a low ratio of concentration, and any large proportion of malachite results in a marked decrease in the percentage of copper recovered.

ADAPTATION OF MINING METHODS TO GEOLOGY AND TOPOGRAPHY.

The methods of prospecting and mining adopted in the Santa Rita district have been determined by the geologic features of the ore deposits, and the general scheme of excavation, ore delivery, and waste disposal has been nicely adjusted to the topographic situation.

By reason of the wide extent of the ore bodies as compared with their depth, they were well suited to churn-drill prospecting for the determination of ore tonnages and grades of ore. The altered an generally soft nature of the rocks resulted in low drilling costs, an the relatively even grade of the ore favored accuracy in determinin the copper content of the ores.

The feasibility of open-cut mining by means of steam shovel depends on the size of the ore bodies and the thickness of the overlying waste.

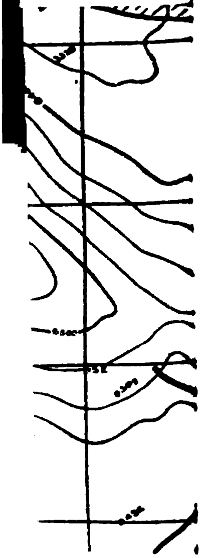
Because the altered rocks that contain the ore and constitute the gangue are soft, drilling is easy. These rocks are much jointed and break readily when blasted.

Any appreciable addition to the amount of waste material that must be moved, owing to large slides, is not anticipated. In general, the overburden of soil, or unconsolidated débris, is shallow, and everywhere such material is essentially free from percolating water. Overburden consisting of weathered rock is also well drained, and both materials stand well without "slaking" in any important degree. Although the ore and the weathered rock contain abundant sericite, and when finely broken develop much colloidal material, in place they are generally strong enough to stand with fairly steep slopes for all depths that are reached by open pits.

The mines are situated in a basin which is a broadened section of the valley of Santa Rita Creek.

In plan the outline of the Santa Rita mine workings is like that of a jew's-harp, two great cuts forming the flaring loop, the block left to carry the creek forming the tongue (see Pl. III). The creek flows southwest, leaving the ore-bearing area 4,000 feet beyond the point of entry and at an elevation 120 feet lower. The inclined approaches that permit locomotive haulage in removing the ore from the deep pits have grades opposed to the general surface slope. These approaches diverge at a point more than 1,200 feet down stream from the westerly edge of the ore area. The length of haul from the far end of each of the mine pits to the common track that serves the crushing plant is thus greater than 1 mile, so that considerable depths can be directly attained. Eventually spirals or switchbacks will be required and possibly the use of hoisting planes or Shay geared locomotives may be advantageous. At present the development plans of the company do not contemplate that any mining will be done by means of steam shovels below an elevation of 6,050 feet, which is approximately 150 feet below the points where the pit approaches intersect the ground surface.

The crusher is situated near the mouth of the canyon-like valley which drains the Santa Rita basin, the delivery track being at a level about 80 feet above the assembly yard. From this yard the ore trains go to the concentrating plant at Hurley by way of the Atchison, Topeka & Santa Fe Railroad.



and the track

Whereas the ore tracks necessarily converge for the purpose of delivery, the waste tracks are in the main divergent. The principal dumping ground is in the valley of Whitewater Creek, south of the mines, but most of the waste from the upper levels of the northern section has been deposited within the Santa Rita basin west and northwest of the mines.

Because of the semiarid character of the climate, current ground-water seepage is slight in comparison with the extent of the workings and pumping costs are correspondingly low. At times of heavy rains the lowest levels in the pits become flooded, but little time is lost from this cause. Continued rains, which are seldom experienced, result in reduced operating efficiency, mainly by causing track troubles.

For some time to come a channel for the creek will be maintained directly across the Santa Rita basin, but eventually, in order to remove large amounts of ore, the diversion of the stream will be necessary. Occasionally after heavy rains the volume of water carried by the creek is large, but the mine pits have been so planned that they are in no danger of being flooded from this source.

The Chino company has, for convenience of reference, grouped its property into six ore bodies, as follows: C. Y. B., Sierra, Hearst, R. W. T., Old Fort, and Estrella. This grouping, however, is somewhat artificial and the divisions do not correspond in every case with actual breaks in the mineralized area. In fact some of the ore bodies are somewhat merged with each other and some are separated by areas of mineralized but low-grade rock. Their annular grouping around a relatively barren core, their extent, and their general relations are shown by Plate III.

HISTORY OF MINING IN SANTA RITA DISTRICT.[a]

It is said that copper was first mined at Santa Rita by the Aztecs. It was probably one of the places where they procured the native metal. About 1800 a friendly Apache chief showed the croppings near the present Romero shaft to Col. José Manuel Carasco, the officer in charge of the Spanish military posts in New Mexico. Col. Carasco interested Don Manuel Francisco Elguea, of Chihuahua, a wealthy merchant banker and subdelegate to the Spanish Court, and the latter obtained a concession to the property under Mexican law. Four years later Elguea purchased Carasco's interests and began to open up the property. The copper produced found a ready market with the Mexican Government for coinage. Almost until the early nineties the region was infested with warlike Apache Indians, who were not subjugated until almost exterminated; hence the Spaniards had to build a fort and maintain a military force to protect the camp. The fort was a triangular structure with heavy built adobe towers guarding each of its apices, and these were joined by strong adobe walls. One of the old towers is still standing and until recently was used as a jail. Other remnants of these ancient fortifications are still extant.

---

[a] The chief source of information regarding past conditions is a manuscript report by Mr. John M. Sully, manager of the Chino Copper Co.

Rough smelting works were built and from the sulphide and oxide ores mined copper metal was extracted in a crude way. This was carried on mule trains, under military convoy, to Chihuahua and on to Mexico City. It is said that Elguea left at his death, in 1809, a considerable fortune. Later, his widow leased the property to Juan Onis. After a few years it was leased to Siqueiros, and was worked intermittently, owing to Indian outbreaks, until the late fifties, when Indian hostility finally forced its abandonment.

Gen. Sibley, in command of Confederate forces from Texas, held this region for a short time. Efforts, mostly unsuccessful, were then made to work the mines.

In 1873 the dawn of a more prosperous period was at hand, for M. B. Hayes, connected with the first smelting works in Colorado, began work at the present Romero mine. A 248-foot shaft was sunk, and a furnace, which proved inadequate, was erected. A shipment of 40 tons of picked ore and imperfectly smelted copper was hauled by teams 700 or 800 miles to a railroad station in Colorado and forwarded to the Baltimore Copper Works and to the Revere Copper Works, at Point Shirley, Mass. In October, 1873, Mr. Hayes and associates obtained title to, and possession of, the property from the sole surviving heirs of the Elguea family, who were settled through Mexico and Europe. Until the heirs had transferred their interests the United States Land Office had refused to issue patent to the property, because the Elguea family had rights under the treaty between the United States and Mexico. Later, patent was taken out by the Hayes interests, so as to make the title more secure and to guard against future litigation.

In 1881 the property was sold to J. Park Whitney, and a stamp mill, later burned, was erected near the foot of Romero Hill. In 1882 diamond drilling was done. Although the records of some of these holes are still extant, their exact location has not been ascertained.

The year 1891 saw the railroad completed to Hanover, a few miles away, and in 1899 a branch into Santa Rita was finished.

In 1897 Mr. Whitney gave a lease and bond to the Hearst estate, but in 1899 sold the property to the Santa Rita Mining Co., before the lease had expired. The new company developed some large bodies of high-grade sulphides and considerable native copper. After extraction of these, active operations were carried on, mostly by lessees, on a basis of 35 per cent to the owners when copper was 15 cents or more per pound, and 25 per cent when it was less than 15 cents. The Santa Rita company, however, continued to operate the mill, treating ore purchased from the lessees and low-grade ore from the dumps, formerly thrown out as waste. It is said that this old mill, which had a capacity of only 120 tons per day, left copper in the tailings to the amount of 4 per cent of the rock treated. It is therefore a far cry from the old methods to present-day milling efficiency. Lessees continued work until about 1906. Ore containing 10 per cent copper was sold by them, but ore with a lower copper content they threw on the waste dumps.

A new era dawned on this ancient mining district in 1906, for in that year systematic sampling of the property was begun by Mr. John M. Sully, the present manager of the Chino company. The old dumps and the old workings were sampled, and churn-drill sampling was begun.

PRESENT CONDITIONS AND EXTENT OF PROPERTY.

After an extensive report by Mr. Sully concerning the results of the sampling mentioned, the present company acquired the property, which now consists of 147 mining claims, including fractional and full claims, comprising 2,645 acres; 131 of these, aggregating 2,412 acres, are patented. In addition, the company owns 160 acres of agricultural land adjacent to its mining claims. The property contained 19 different shafts, aggregating over 4,000 feet of sinking, and these were connected by several thousand feet of underground workings.

This, then, was the beginning of the development as a result of which an old and inefficient mill that treated 120 tons of ore per day has been replaced by a modern mill, with a capacity of about 7,000 tons per day, which successfully treats ore containing less than 0.8 per cent of copper.

Stripping operations were started by the Chino company in October, 1910, the ore taken out being sent to stock piles. During August, 1911, the first shipment of ore was made to the mill. The total shipments during that month were nearly 14,000 wet tons.

PROSPECTING AND LOCATING ORE BODIES.

Preliminary to the systematic prospecting and delimiting of the ore bodies with churn drills, an accurate topographic map of the region, on a scale of 100 feet to the inch and showing a contour interval of 5 feet, was made. This map was then laid off into north-south and east-west coordinates at intervals of 100 feet, and a datum point at a coordinate intersection was established, for the purpose of locating, by coordinates referred to this point, all the drill holes that were to be put down, as for instance: Hole 10, north 650 feet and west 355 feet.

Bulletin 107

DEPARTMENT OF THE INTERIOR
FRANKLIN K. LANE, SECRETARY
BUREAU OF MINES
VAN. H. MANNING, DIRECTOR

PROSPECTING AND MINING OF COPPER ORE AT SANTA RITA, NEW MEXICO

BY

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WASHINGTON
GOVERNMENT PRINTING OFFICE
1916

Form 1.—*Form used by driller in reporting to mine office the work done by his crew.*[a]

DAILY CHURN-DRILL REPORT.

SAMPLES.

Depth.	Character of rock.		Minerals.	Sampling appliance.		Caving.			Is sample reliable? If not, state reasons under "General remarks."	True depth.	Length of sample.
Number of sample.	1. Quartz-diorite. 2. Andesite-porphyry. 3. Quartzite. 4. Limes or other rock. Leached or unleached. Altered or unaltered. Hard or soft. 5. Silicified porphyry. 6. Central city intrusive.	Color of sludge: Red or dark brown. Light yellow. Pink or purple. Light brown. Gray or white. Green or blue.	A Chalcocite. B Pyrite. C Metallic copper. D Oxides of copper. E Carbonates. F Chalcopyrite. G Magnetite. H Hematite. I Mica.	Split divider.	Tub.	No.	Yes; slightly.	Yes; considerably.			

GENERAL REMARKS.

....

....

....

Water.—Depth reached............ Stands at depth of..........................

....

Faults and Clay Seams.—Depth below surface...ft. Width of fault material...ft.
Size of Bit.......... Casing: Size..........; depth..........

EMPLOYMENT OF TIME.

Actual drilling hours..................... Moving and setting up hours............
Repair hours............................ Casing or removing casing...............
Other delays...
Driller..................................... Shift...........................
Helper..................................... Date...........................
Sampler.................................... Hole No. ..... Drill No. .....

Note.—Enter time after each name.

---

[a] Size of form as printed by operating company, 8½ by 13½ inches; held in binder through two holes in top.

In the office the data from the daily drillers' reports were compiled into a monthly report (Form 2). The average performance and efficiency of the churn drills used in prospecting the Santa Rita ore bodies can best be shown by the table following. In this the performance of two Cyclone No. 6 deep-hole drills is averaged for a period of 10 months. The figures are fairly representative of what has been accomplished by prospecting drills on the somewhat softened and locally sheared granitic and other rocks of Santa Rita. A similar table, showing in a comparative way the performance of all the drills, was made out for each month.

FORM 2.—*Form*[a] *used in compiling monthly report from data in daily drillers' reports, as shown in Form 1.*

Date.	Shift.	Hole.	From—	To—	Number of feet drilled.	Hours drilling.	Hours moving.	Hours casing.	Hours reaming.	Hours repairing.	Other delays.	Total time.	Drill No.	
													Driller.	Helper.
1														
2														
3														
4														
5														
6														
7														
8														
9														
10														
11														
12														
13														
14														
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18														
19														
20														
21														
22														
23														
24														
25														
26														
27														
28														
29														
30														
31														
Totals														

---

[a] Size of form used by operating company, 8½ by 14 inches; upper half only, showing space for recording data covering day shift, is shown; lower half provides for similar data covering night shift. Held in binder through four holes at left.

Churn-drill record.

[March to December, 1912—10 months.]

Drill No.	Feet drilled.	Total working hours.	Per cent time drilling.	Per cent time delayed.	Per cent time repairing.	Per cent time moving.	Average feet per hour of drilling.
DAY SHIFT.							
6	3,221	3,727	59.3	18.2	3.3	19.2	1.46
7	3,365	3,776	70.7	10.6	2.5	16.2	1.26
NIGHT SHIFT.							
6	3,071	2,598	78.9	16.4	3.5	1.2	1.50
7	2,947	2,850	84.4	12.3	2.2	1.1	1.23
TOTAL.							
6	6,292	6,325	67.0	17.5	3.4	12.1	1.48
7	6.312	6,626	76.4	11.3	2.4	9.9	1.25

COST OF PROSPECT DRILLING.

To the miner the cost of prospecting is of great importance; hence cost data regarding churn drilling these ore bodies are, through the courtesy of the Chino Copper Co., presented in some detail, as follows:

Cost data on churn or prospect drilling at Santa Rita, N. Mex.

Drill Nos.	Month.	Cost of—						
		Labor.	Supplies.	Teaming.	Administration.	Total.	Per foot.	Per shift.
	1912.							
6, 7, and 12.	May	$2,106.14	$494.03	$142.18	$349.28	$3,091.63	$2.31	$22.90
6 and 7	June	2,029.35	661.09	80.25	270.23	3,040.92	2.29	26.44
6 and 7	July	1,997.43	451.20	112.75	229.67	2,791.05	2.23	24.48
6 and 7	August	1,969.57	496.14	129.00	209.12	2,803.83	2.00	23.56
6 and 7	September	1,953.78	442.02	150.25	234.24	2,780.29	2.13	24.56
6 and 7	October	1,823.99	506.98	153.95	185.25	2,670.17	2.68	23.95
6 and 7	November	1,775.52	619.47	134.75	238.54	2,768.28	2.42	27.82

The above table is valuable as a general index of the cost of churn-drill prospecting. Of course, in other places local conditions might cause considerable variation from the figures given.

ASSAY RECORDS AND ORE CLASSIFICATION.

The assay returns were compiled on loose leaves (Form 3), which, when filled out, were put into strong permanent binders and kept as volumes. In the columns of this form the location of the sample in the hole was recorded under depth and its value when assayed by the permanganate method was shown in the "Perm. Cu." column.

FORM 3.—*Form used in assay record of drill-hole samples.*[a]

DRILL HOLE NO. ....

Coordinates.........................

Time drilling........................

Depth.	% Perm. Cu.	% Met. Cu.	% Cu. total.	% Elect. Cu.	Averages.	Rock.	Minerals.	Sludge.

The results of electrolytic check assays were recorded under the heading "Elect. Cu." In the column headed "Averages," the ore was divided into classes, as follows:

Ore classification used by Chino Copper Co.

Class.	Copper content, per cent.	Designation.
P	0.00 to 0.79	Poor.
L 1	0.80 to 0.89	Low No. 1.
L 2	0.90 to 0.99	Low No. 2.
F	1.00 to 1.24	Fair.
G	1.25 to 1.49	Good.
E	1.50 to 1.99	Excellent.
S	2.00 upward	Superlative.

Any native copper contained was recovered by screening or panning the crushed sample; this was weighed and the weight recorded in the column headed "Met. Cu." The sum of the native copper content and the copper determined by the permanganate method was then set down in the column headed "Cu. total." Notes on the kind of rock passed through, the minerals encountered, and the color and character of the sludge were set down in the columns headed "Rock," "Minerals," and "Sludge." It will be seen that this form gave a comprehensive record of each hole, including its location by coordinates, time of drilling, depth, values for every assay interval of 3 feet, and averages. However, to more thoroughly visualize the mineralized conditions indicated in each hole, its assay record was plotted on cross-section paper somewhat as shown on Form 4. Two ink lines to represent each drill hole were drawn as shown. The sections as determined by the groups of assay results were indicated by horizontal ink lines, as shown, each small square on the paper representing a depth, or sampling interval, of 3 feet; the figures opposite showed the percentage of copper that the rock contained.

In grouping the ore into the foregoing classes, every sudden and considerable change in values, if the record showed a change in the character of the sludge, was noted as a possible class boundary. Rock carrying less than 0.8 per cent of copper was considered of too low grade to be classed as ore, as is explained later. However, where small masses of waste occurred with the ore, in such a way that separation from the latter could not profitably be carried out, it was figured in as a diluent of the ore reserves.

CROSS-SECTIONING THE ORE BODIES.

Following the assay-record sheets came the work of making cross sections of the ore body. These were compiled from the data shown on the assay records and the drill hole records and show both north-south and east-west cross sections of the ore bodies at intervals of 100 to 200 feet, depending on the spacing of the drill holes and the data that they furnished. Where the holes were several feet away from a cross section, the data from them were projected to the section and recorded there, with a statement of the distance and direction of the hole from the section. These cross-section sheets are used to show the general form and relations of the ore body and the relations of the different classes of ore with the barren areas and with each other.

A typical generalized cross section is shown in Form 5. The scale is the same as that used by the Chino company, namely, 1 inch equals 200 feet horizontally and 60 feet vertically. Each hole shows in place the number of feet of each class of ore, and of waste, that is encountered. Lines were drawn connecting points of approximately equal values; thus, by the law of averages, the relative quantities of each class were graphically shown on the section.

The figures *20*, *21*, *23*, etc., along the top of Form 5 are the numbers of the drill holes along the line of the cross section, the cross section being constructed from the data revealed by these holes. The numbers 2′ N, 3′ S, etc., under some of the hole numbers mean that the hole is 2 feet north, or 3 feet south of the actual cross-section line, but the data from holes that are so close to the section are projected onto the section just as though the holes were in line. The P areas (unshaded) show the relations of waste material to ore, etc. The shaded areas that lie inside and above the heavy dotted line are ore areas, and these are divided into two different classes by two different patterns of shading. The shaded areas are further subdivided into a total of five classes of ore by light dotted lines connecting points of similar change of value in each drill hole.

The ore below the 6,050-foot level can probably not be recovered by steam-shovel mining and the present methods of rail haulage, so that the cost of recovering it will be somewhat increased.

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The notation 10 N., East and West crosssection, at the top of the cross section means that this is an east and west cross section, and is the tenth north from cross section 1. The figures 1000, 1500, etc., are the horizontal distances in feet. The vertical distances, referred to sea level, are shown by the figures 6050, 6200, 6350, etc., on the right margin.

The use of colors to denote the different classes of ores shown by the cross section adds greatly to its clearness. In the form as used at the mine two colors, indicated by shading in the form as presented, were used to bring out the special relations.

Figure 2 represents two drill holes. Hole *1* shows 3 feet of class E ore below 17 feet of barren rock. Hole *2* shows 10 feet of class E ore and 10 feet of class S ore below 10 feet of barren rock. A straight (dotted) line connects the top boundary of class E ore in hole *1* with the top boundary of this class of ore in hole *2;* and similarly for the bottom boundary. Although no class S ore was found in hole *1* yet the class S ore body found in hole *2* may maintain its full thickness out nearly to hole *1*, so, by the law of averages, it is approximately correct to figure the ore as occupying the triangular space C.

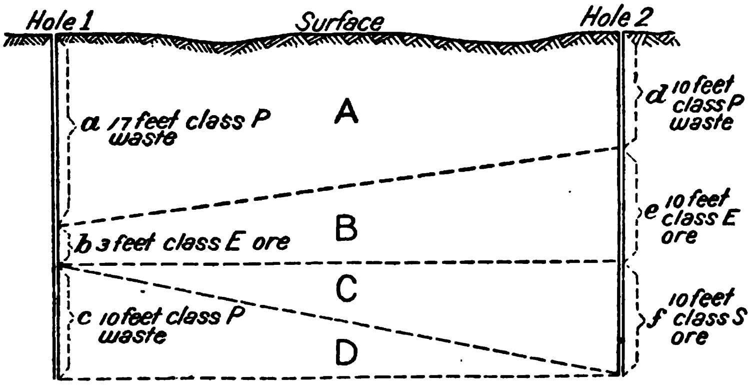


FIGURE 2.—Method of estimating classes of ore from drill records so as to show them on cross sections. *a*, hole *1*, 17 feet of class P rock or waste; *b*, 3 feet of class E ore; *c*, 10 feet of class P rock; *d*, hole *2*, 10 feet of class P rock; *e*, 10 feet of class E ore; *f*, 10 feet of class S ore. The area A would be waste, the area B would be class E ore, and the triangular area C would be S ore. It would be figured as extending up to hole *1* though no S ore had actually been encountered in that hole. The D area would be waste rock that would not be moved.

Cross sections are very helpful in showing the trend of ore bodies and their form and general relations. Where geological conditions can be shown on the cross sections they are especially valuable in studying ore deposits. Such cross sections as are presented are not quite accurate enough for use in making final estimates of ore tonnages. A more accurate method of ore estimation is discussed in a subsequent section.

GENERAL CONSIDERATIONS IN DELIMITING ORE BODIES.

An inspection of the completed cross-section sheets made fairly evident just what ground could profitably be mined under the conditions. With the cross-section data as a basis, the limiting outlines of the ore bodies were drawn on maps showing the surface configuration, drill holes, etc. The chief considerations that governed the mapping of the horizontal limits to which ore could be mined were as follows: (1) The depth of the overburden and the slopes at which its banks would stand; (2) the local size and thickness of the ore body and its character and value; (3) the method of mining—whether the ore body could all be taken out by steam shovels at about the same average cost as the main part of the ore body, or whether extra expense would be involved.

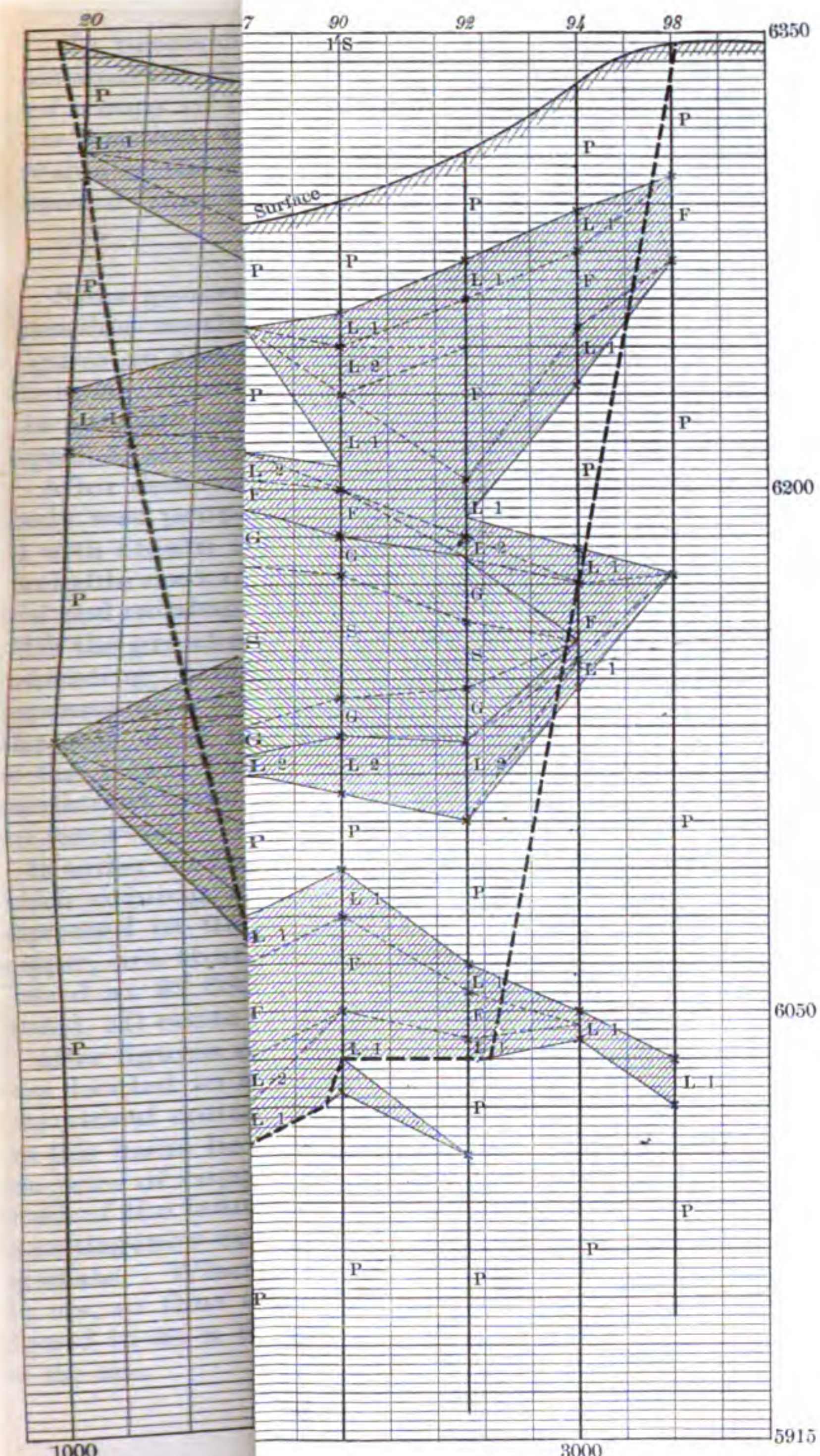
Of course, it was recognized that there must necessarily be a limit to the depth to which steam-shovel mining with locomotive haulage could be carried. After consideration of various factors it was believed that below the 6,050-foot level (measured from sea level) the great open-cut mining pits would be so deep and so restricted in area at the bottom that certain modifications in the methods of mining the ore below that level—a relatively small percentage of the total ore—would have to be adopted. The chief factors regulating the depth to which it would be convenient to mine with steam shovels were believed to be as follows: (1) The horizontal extent of the ore body, in relation to its depth, would have to be considered because a certain amount of "elbow room" would be necessary for steam shovels, car tracks, etc.; (2) the maintenance of economical transportation grades for hauling out the ore and waste would limit the depth of working by steam shovels and locomotive haulage; (3) if the horizontal dimensions of the deposit were great enough, steam shovels might load the ore even in very deep pits, and it might be hauled out by means of Shay or rack-geared locomotives, or over inclined planes with cable haulage.

Estimates of the ore above the 6,050-foot level were kept separate from those of the relatively small proportion of ore below that level, as the cost of taking out the lower ore would be slightly greater.

METHODS OF ESTIMATING ORE.

When the facts collected in prospecting and sampling the ore bodies were supplemented by geologic and by general mining cost data, the whole body of information thus systematically gathered was used in estimating the tonnage of rock rich enough to be classed as ore and available for mining.

grades of ore, are explained on page 15.]



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ASSAY PLAN.

In proceeding toward a methodical solution of the problem of ore estimation the first step was to make an assay plan. This consisted of a copy of the original drilling map showing each hole, by number, its location, the number of feet of ore encountered in it, the average value of the ore, the number of feet of waste that would have to be moved from the ore there, etc.

PRELIMINARY ESTIMATES.

Some general idea of the form and size of the ore body, as well as possible methods of working it, had to be obtained before definite figures on the amount of minable ore available could be made. For instance, if only a quarter of a million tons of ore was in the deposit, the average cost per ton for mining would be more, other things being equal, than if it contained 5,000,000 tons.

After determination had been made, in a general way, that the ore body was properly situated and extensive enough to warrant mining it with steam shovels on a large scale, an estimate was made of the probable cost of removing and dumping the valueless stripping material and mining the ore by open-cut methods on a scale commensurate with the great size of the deposit. The estimated cost, including concentrating, smelting, marketing the product, all wastage, and all expenses was $2 per ton for moist ore. From the cost data on mining, shipping, and treating the ore from the somewhat similar ore bodies of the Utah Copper Co.'s properties, it was known that the figure mentioned was a conservative one on which to base estimates of the ore reserves.

In order to facilitate the estimation of ore, the critical worth, below which mining at a profit could not in general be counted on, was expressed in units or values of copper. At 13 cents per pound for copper, ore that assayed 0.8 per cent, or 16 pounds of metal per ton, valued at $2.08, meant at least 8 cents per ton profit, if, as above stated, all costs and wastage allowances amounted to $2 per ton.

After figures of this kind had been prepared in this general way, it was decided to adopt 0.8 per cent of copper as what might be called the critical content. All rock containing a less percentage could not on this basis be estimated as ore. However, it was realized that if the price of copper went up considerably, then wherever practicable much of the leaner rock would undoubtedly be mined as ore, but such a contingency could not, of course, be made a factor in a conservative estimate. Wherever a mass of lean rock happened to be surrounded by ore, so that separating it was unprofitable, then of course it was figured in as a diluent, which reduced somewhat the average tenor of the ore.

A critical copper content of 0.8 per cent having been fixed as the content below which the rock could not conservatively be called ore, there being very little gold or silver in it, the estimate was made first by the cross-section method, which consisted in estimating the mass of ore by means of square prisms and then by figuring its value from the drill-hole and cross-section data. Later, as a check, the more exact triangular-prism method was used. As this method is better adapted for close and careful ore estimation, though involving about the same general principle as the cross-section method, it is explained in detail below.

ESTIMATING ORE BY TRIANGULAR-PRISM METHOD.

From the assay record of each drill hole the ore reserves were calculated. For these calculations three well-bound books with blank pages were used. They were each about 9½ by 12 inches in size and were cross ruled on a scale of 4 spaces to the inch. Book 1 may be called the log book of prospect drill holes. On page 1 were recorded the ore calculations for hole 1, on page 2 the calculations for hole 2, and so on.

The following examples are to show how the drill-hole records, prepared on Form 4, were calculated in Book 1:

Specimen calculation sheets from drill-hole records shown in Form 4.

HOLE A (E. 100; S. 0.00.).

LOG OF HOLE.

Feet of ore.	Class.	Per cent copper.	Foot per cent.
19	P[a]	.....	......
9	L 2	.907	8.16
15	F	1.186	17.79
6	L 1	.875	5.25
21	S	2.114	44.39
15	E	1.880	28.20
60	P[a]	.....	......
6	L 1	.850	5.10
15	G	1.480	22.20
9	L 1	.817	7.35
12	E	1.500	18.00
51	S	2.600	132.60
12	F	1.150	13.80
15	L 1	.840	12.60
54	P[a]	.....	......
9	L 1	.883	7.94
12	E	1.700	20.40
21	S	2.157	45.30
12	L 2	.912	10.94
15	F	1.160	17.40
12	L 1	.838	10.05
60	P[a]	.....	......
12	F	1.158	13.89
27	S	2.135	57.64
12	F	1.112	13.34
9	L 1	.850	7.65
520			519.99

ORE OF CLASS S.

Feet of ore.	Per cent copper.	Foot per cent.
21	2.114	44.39
51	2.600	132.60
21	2.157	45.30
27	2.135	57.64
120		279.93

ORE OF CLASS E.

15	1.880	28.20
12	1.500	18.00
12	1.700	20.40
39		66.60

ORE OF CLASS G.

15	1.480	22.20

ORE OF CLASS F.

15	1.186	17.79
12	1.150	13.80
15	1.160	17.40
12	1.158	13.89
12	1.112	13.34
66		76.22

ORE OF CLASS L 2.

9	.907	8.16
12	.912	10.94
21		19.10

ORE OF CLASS L 1.

6	.875	5.25
6	.850	5.10
9	.817	7.35
15	.840	12.60
9	.883	7.94
12	.838	10.05
9	.850	7.65
66		55.94

CLASS P WASTE.

193	.....	......
520		519.99

---

[a] Waste.

HOLE B (E. 210; S. 0.00.).

LOG OF HOLE.

Feet of ore.	Class.	Per cent copper.	Foot per cent.
33	P[a]	.....	......
12	L 2	.937	11.24
9	F	1.133	10.19
12	E	1.750	21.00
18	S	2.283	41.09
6	F	1.050	6.30
12	E	1.935	23.22
15	L 1	.810	12.15
12	F	1.075	12.90
69	P[a]	.....	......
18	L 1	.861	15.49
15	F	1.204	18.06
9	S	2.077	18.69
12	L 2	.950	11.40
18	G	1.266	22.78
15	E	1.920	28.80
24	L 2	.924	22.18
48	P[a]	.....	......
12	L 1	.887	10.64
15	G	1.320	19.80
15	S	2.100	31.50
12	E	1.775	21.30
9	L 2	.950	8.55
57	P[a]	.....	......
12	L 1	.850	10.20
9	F	1.233	11.10
15	S	2.100	31.50
6	G	1.300	7.80
9	L 1	.883	7.95
528			435.83

ORE OF CLASS S.

Feet of ore.	Per cent copper.	Foot per cent.
18	2.283	41.09
9	2.077	18.69
15	2.100	31.50
15	2.100	31.50
57		122.78

ORE OF CLASS E.

Feet of ore.	Per cent copper.	Foot per cent.
12	1.750	21.00
12	1.935	23.22
15	1.920	28.80
12	1.775	21.30
51		94.32

ORE OF CLASS G.

Feet of ore.	Per cent copper.	Foot per cent.
18	1.266	22.78
15	1.320	19.80
6	1.300	7.80
39		50.38

ORE OF CLASS F.

Feet of ore.	Per cent copper.	Foot per cent.
9	1.133	10.19
6	1.050	6.30
12	1.075	12.90
15	1.204	18.06
9	1.233	11.10
51		58.55

ORE OF CLASS L 2.

Feet of ore.	Per cent copper.	Foot per cent.
12	.937	11.24
12	.950	11.40
24	.924	22.18
9	.950	8.55
57		53.27

ORE OF CLASS L 1.

Feet of ore.	Per cent copper.	Foot per cent.
15	.810	12.15
18	.861	15.49
12	.887	10.64
12	.850	10.20
9	.883	7.95
66		56.43

CLASS P WASTE.

Feet of ore.	Per cent copper.	Foot per cent.
207	.....	......
528		435.83

---

[a] Waste.

HOLE C (E. 190; S. 130.).

LOG OF HOLE.

Feet of ore.	Class.	Per cent copper.	Foot per cent.
42	P[a]	.....	......
18	L 1	.882	15.88
21	G	1.263	26.52
12	E	1.837	22.04
21	F	1.085	22.79
6	E	1.975	11.85
6	G	1.300	7.80
48	P[a]	.....	......
12	L 2	.912	10.94
9	G	1.333	12.00
12	E	1.850	22.20
24	S	2.150	51.60
6	E	1.775	10.65
15	F	1.160	17.40
12	L 1	.832	9.98
18	F	1.120	20.16
12	E	1.837	22.04
12	F	1.100	13.20
12	L 2	.950	11.40
60	P[a]	.....	......
9	L 2	.910	8.19
15	F	1.154	17.31
15	E	1.880	28.20
21	S	2.128	44.69
18	E	1.600	28.80
30	P[a]	.....	......
6	L 2	.900	5.40
39	G	1.397	54.48
531			495.52

ORE OF CLASS S.

Feet of ore.	Per cent copper.	Foot per cent.
24	2.150	51.60
21	2.128	44.69
45		96.29

ORE OF CLASS E.

Feet of ore.	Per cent copper.	Foot per cent.
12	1.837	22.04
6	1.975	11.85
12	1.850	22.20
6	1.775	10.65
12	1.837	22.04
15	1.880	28.20
18	1.600	28.80
81		145.78

ORE OF CLASS G.

Feet of ore.	Per cent copper.	Foot per cent.
21	1.263	26.52
6	1.300	7.80
9	1.333	12.00
39	1.397	54.48
75		100.80

ORE OF CLASS F.

Feet of ore.	Per cent copper.	Foot per cent.
21	1.085	22.79
15	1.160	17.40
18	1.120	20.16
12	1.100	13.20
15	1.154	17.31
81		90.86

ORE OF CLASS L2.

Feet of ore.	Per cent copper.	Foot per cent.
12	.912	10.94
12	.950	11.40
9	.910	8.19
6	.900	5.40
39		35.93

ORE OF CLASS L 1.

Feet of ore.	Per cent copper.	Foot per cent.
18	.882	15.88
12	.832	9.98
30		25.86

CLASS P WASTE.

Feet of ore.	Per cent copper.	Foot per cent.
180	.....	......
531		495.52

---

[a] Waste.

In explanation of the calculation sheets shown it may be said that the number of the hole and its location by coordinates are set down at the top of the page, as Hole A (E. 100; S. 0.00). In the left-hand column the number of feet of each class of ore or waste, according to the classification mentioned on page 15, is shown in proper relative position, having been taken direct from the assay record of the drill hole (Form 4). The average assay of each class of ore in percentage of copper is shown in the second column.

Now 10 feet of 1 per cent ore and 20 feet of 2 per cent ore would not give 30 feet of 1.5 per cent ore, but 30 feet of 1.667 per cent ore. This correct average is obtained by taking account of the fact that there is twice as much of the rich 2 per cent ore as of the 1 per cent ore. The calculation follows:

Feet of ore.		Per cent copper.		Foot per cent.
10	×	1	=	10
20	×	2	=	40
30				50

Dividing the total foot per cent, 50, by the total feet of ore, 30, gives 1.667 per cent of copper as the average value throughout the 30 feet.

In like manner the number of feet of each class of ore is multiplied by its assay value, and the product is set down under the column marked "Foot per cent." The depth of the hole to the bottom of the lowest ore divided into the total feet per cent would give the average value for that hole. On the right-hand side of the page are segregated the figures for the different classes of ore for the given hole, the number of feet of each class, and its foot per cent.

Book 2, used in the ore calculations, may be called the "triangular-prism book." In its records the ore body is regarded as composed of a series of vertical triangular prisms, and the ore grade of each prism is separately calculated. The first step in this calculation is to take a copy of the drill-hole map and connect, by straight lines, each drill hole with its nearest neighbors, in such a way that the whole ore-bearing area is divided into triangles, with the apices of each triangle, or rather the edges of each triangular prism, marked by a drill hole. The ore grade in each triangular prism is thus closely approximated by the average grades found in the three drill holes forming its edges.

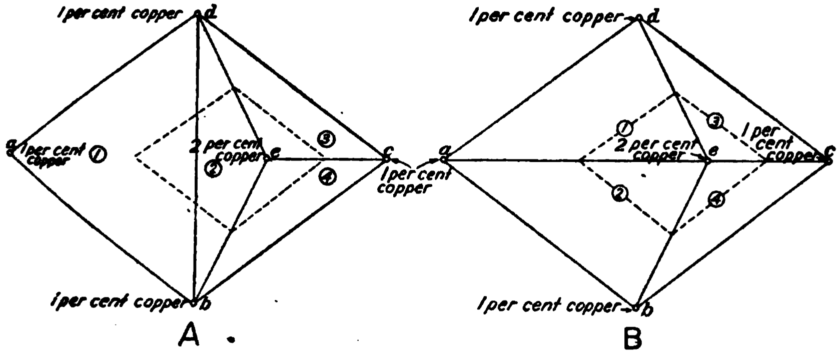

FIGURE 3.—Methods of dividing ore area into triangles. A, incorrect method; B, correct method.

There is a right way and a wrong way to lay off these triangles. Suppose five drill holes were placed and showed values about as in figure 3.

Figure 3, A, shows the wrong way to triangulate these holes, if divided in the way shown, triangle *1* would show only 1 per cent of ore, whereas in reality the 2 per cent ore should be calculated as extending halfway from *e* to *a*, as indicated by the dotted lines. Of course, in any particular case, the rich ore might not extend halfway toward *a*, but, according to the law of averages, it should be calculated as though it did, so as to make proper apportionment of the high as well as of the low values. If the drill-hole area were divided as in figure 3, A, triangle *1* would be undervalued and triangle *2* overvalued.

Correct apportionment of the values to each of the triangles is shown in figure 3, B. The data on which to base properly evaluated triangulation are obtained not only from the average assays of each hole, but largely from the geologic information, such as the continuity of the ore values, shown on the cross sections.

When all of the ore-bearing drill holes have been connected by lines so that each hole forms the apex of one or more triangles, each triangle so formed is given a number. On page 1 of the triangle book are found the calculations for triangle 1, as follows:

Specimen page from triangle book.

TRIANGLE 1. AREA, 7,150 SQUARE FEET. HOLES A, B, AND C.

[Data taken from calculations on pages 20, 21, and 22.]

Hole.	Class of ore.												P, feet of waste.
	S.		E.		G.		F.		L 2.		L 1.		
	Feet of ore.	Foot per cent.	Feet of ore.	Foot per cent.	Feet of ore.	Foot per cent.	Feet of ore.	Foot per cent.	Feet of ore.	Foot per cent.	Feet of ore.	Foot per cent.	
A.......	120	279.93	39	66.60	15	22.20	66	76.22	21	19.10	66	55.94	193
B.......	57	122.78	51	94.32	39	50.38	51	58.55	57	53.37	66	56.43	207
C.......	45	96.29	81	145.78	75	100.80	81	90.86	39	35.93	30	25.86	180
a[a].......	222	499.00	171	306.70	129	173.38	198	225.63	117	108.40	162	138.23	580
b[b].......	74	166.33	57	102.23	43	57.79	66	75.21	39	36.13	54	46.08	193.33
c[c].......	529,100		407,550		307,450		471,900		278,850		386,100		......
d[d].......	1,189,260		730,945		413,199		537,752		258,330		329,472		......

[a] Line a shows sum of the number of feet and foot per cent of the different classes of ore for the three holes.
[b] Line b shows one-third of a, or the average of the three holes.
[c] Line c shows average thickness multiplied by area, giving cubic feet of each class of ore in prism.
[d] Line d shows average foot per cent multiplied by area giving cubic foot per cent or "volume assay" of each class of ore in prism.

P.
A=193
B=207
C=180
580

$\frac{580}{3}\times 7{,}150=\frac{4{,}147{,}000}{3}=1{,}382{,}333$ cubic feet, or amount of waste contained in triangular prism 1.

The sample of the calculations set forth in the triangle book shows that the area of the triangle and the number of the holes from which its ore content is calculated are set down at the top of the page. Line a shows the total number of feet of ore and the foot per cent of each class of ore in each hole. The average thickness and the average foot per cent of each class for the prism (one-third of a) are entered in line b. Line c shows the average thickness of each class of ore multiplied by the cross-sectional area of the prism. This gives the number of cubic feet—the volume—of a particular class of ore. The d line shows the cubic foot per cent, or the volume assay, for each class of ore in the triangular prism. It is obtained by multiplying the average foot per cent by the cross-sectional area of the prism.

The third book for ore calculations is of the same type as the other two, and may be called the book of ore totals. In it are recorded the volume of ore and the volume assay, or cubic feet per cent, of each class in each triangular prism for each ore body. Let us assume that ore body A contains 100 triangular prisms of ore, each exactly equal to prism 1, already recorded. The ore in such an ore body would be summed up in the book of ore totals somewhat as follows:

Specimen page from book of ore totals.

TOTAL ORE IN ORE BODY A.

Class of ore.	Cubic feet of ore.	Volume assay or cubic feet per cent.	Per cent copper.	Tons of ore, at 13 cubic feet per ton.
S	52,910,000	118,926,000	2.247	4,070,000
E	40,755,000	73,094,500	1.793	3,135,000
G	30,745,000	41,319,900	1.343	2,365,000
F	47,190,000	53,775,200	1.139	3,630,000
L 2	27,885,000	25,833,000	.926	2,145,000
L 1	38,610,000	32,947,200	.853	2,970,000
	238,095,000	345,895,800	1.453	18,315,000

Dividing the volume assay, or cubic feet per cent, of ore by the cubic feet gives the average assay value, in per cent, for each class and for the total ore.

In order to convert the cubic feet of ore into tons, the average specific gravity of the ore body must be determined.[a] If found to be 2.5, then a cubic foot of ore would weigh 2.5 times the weight of a cubic foot of water, or $2.5 \times 62.5 = 156.25$ pounds; thus 13 cubic feet of such ore would weigh approximately a ton. Thirteen cubic feet of Chino ore in the solid and 21 cubic feet of broken ore were found to average practically a ton, and this average was used in the calculation of ore reserves. Dividing the cubic feet of ore by 13 gives the tonnage shown above—18,315,000 tons of ore, containing an average assay value of 1.453 per cent copper, or practically 266,117 tons of copper. The actual amount of this copper that could be recovered would of course depend on the percentage of recovery made by the mill and smelter.

To summarize, holes were drilled through the ore body every 100 or 200 feet, and these were sampled at depth intervals of 3 feet. The assay values from each sample were arranged in order, and grouped into classes as shown in Form 4. The classes were entered in the log book of prospect drill holes, and the drill holes were grouped together in sets of threes, each set of three marking the edges of a vertical triangular prism. The average values for each prism were calculated in the triangular prism book; and finally the total ore for each ore body was summed up in the book of total ore. These books, with all calculations, are preserved with other company records.

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[a] For diagram and formula convenient for ore calculations see Mead, W. J., The relation of density, porosity, and moisture to the specific volume of ores: Econ. Geol., vol. 3, June-July, 1908, pp. 319-325.

The results from the book of ore totals were arranged into tables as follows: A table of total ore developed up to March 1, 1912, and the yardage of stripping that would have had to be moved to recover it; a table of the total steam-shovel ore developed up to the same date, and the yardage of stripping that covered it; a table of additional steam-shovel ore developed after March 1, 1912. Of these tables the one covering the amount of steam-shovel ore developed up to the date mentioned follows:

Data on amount of steam-shovel ore developed up to Mar. 1, 1912.

Name of ore body prospected.	Steam-shovel ore.																Stripping.[b]		Ratio of ore to waste.
	Class S.[c]		Class E.[c]		Class G.[c]		Class F.[c]		Class L2.[c]		Class L1.[c]		Class P.[c]		Total tons.	Average assay.	Slope.		
	Tons.	Average assay [a]	Tons.	Average assay.	Tons.	Average assay.	Tons.	Average assay.	Tons.	Average assay.	Tons.	Average assay.	Tons.	Average assay.			Cubic yards broken.	Tons.	
C. Y. B.......	3,441,710	2.85	2,617,770	1.67	1,758,932	1.37	2,084,220	1.13	1,034,788	0.95	625,754	0.86	458,342	0.55	12,021,516	1.72	6,527,129	8,392,023	1:0.70
R. T. W......	4,681,412	2.91	3,320,939	1.71	2,219,884	1.37	3,282,042	1.11	1,690,213	.94	1,260,122	.85	916,712	.58	17,371,323	1.68	10,989,870	14,129,833	1:0.81
Hearst........	13,324,098	3.41	4,926,392	1.66	4,429,134	1.35	4,110,055	1.10	2,784,595	.94	2,353,135	.84	1,512,191	.61	33,439,599	2.08	21,028,461	27,036,593	1:0.81
Old Fort.....	31,195	3.30	61,913	1.59	133,116	1.32	76,826	1.12	31,464	.91	6,788	.82	19,616	.48	360,918	1.41	325,180	418,088	1:1.16
Sierra.........	1,930,258	2.71	4,058,965	1.70	2,938,513	1.35	2,895,046	1.10	1,440,814	.93	994,511	.85	699,634	.63	14,957,741	1.46	13,229,328	17,009,136	1:1.14
Estrella......	1,120,984	2.66	1,206,484	1.69	555,521	1.35	858,734	1.10	210,882	.94	193,593	.85	242,991	.51	4,389,190	1.64	9,367,943	12,044,498	1:2.74
Total...	24,529,657	3.14	16,192,463	1.68	12,035,100	1.36	13,306,923	1.11	7,192,756	.94	5,433,908	.85	3,849,486	.59	82,540,287	1.81	61,467,911	79,030,171	1:0.96

[a] Each average assay is expressed as per cent of copper per ton of ore.
[b] The slope at first was ½ horizontal to 1 vertical, but in 1915, after the stripping had been nearly completed, a slope of ¾ to 1 was adopted.
[c] For explanation of ore classification, see p. 15.

FORMULAS FOR ESTIMATING ORE.

FORMULA FOR DETERMINING LOWEST GRADE OF ROCK THAT MAY BE CLASSED AS ORE.

The formulas following may be largely credited to Mr. L. E. Foster, assistant superintendent of the Chino company's mine. The authors merely developed a little further his fundamental formula. Formula *1* is for use in determining what grade of rock might be mined as ore under varying conditions of costs and of copper prices. The factors in the equation are explained below. Certain cost figures are given to be substituted in the equations. These figures, however, are being modified by more efficient methods as time goes on.

$$\frac{M+F_0+m}{AC\times2000\times0.95}+\frac{f_0+S}{D\ 2000\times0.95}+R+E+c=B\frac{100}{105} \tag{1}$$

Simplifying equation *1* we get:

$$\frac{M+F_0+m}{1900\ C}+\frac{A\ (f_0+S)}{1900\ D}+A(R+E+c)=AB\frac{100}{105} \tag{2}$$

Transposing equation *2* gives:

$$\frac{M+F_0+m}{1900\ C}=A\left[\frac{100\ B}{105}-\frac{(f_0+S)}{1900\ D}-(R+E+c)\right] \tag{3}$$

Transposing equation *3* gives:

$$A=\frac{\dfrac{M+F_0+m}{1900\ C}}{\dfrac{100\ B}{105}-\dfrac{(f_0+S)}{1900\ D}-(R+E+c)} \tag{4}$$

Simplifying equation *4* gives:

$$A=\frac{M+F_0+m}{1900\ C\left[\dfrac{100\ B}{105}-\dfrac{(f_0+S)}{1900\ D}-(R+E+c)\right]} \tag{5}$$

Values substituted for factors in foregoing equations.

Factor.	Definition of factors.	Value.
$M=$	Mining costs per dry ton of ore	$0.4683
$F_0=$	Freight from mine to mill per dry ton of ore	.1111
$m=$	Milling costs per dry ton of ore	.5776
$f_0=$	Freight on concentrates per dry ton from mill to smelter	1.1191
$S=$	Smelting charges per dry ton of concentrates	6.352
$R=$	Refining and delivering charges per pound of copper paid for	.0106
$E=$	Export freight and insurance charges per pound of copper paid for	.0014
$c=$	Commission on sales	$\frac{1}{100}B$
$B=$	Value per pound of copper	.13
$C=$	Per cent of extraction by the mill	65
$D=$	Per cent of copper in the concentrates	25
$A=$	Per cent of copper per dry ton of ore.	

The constant 2,000 stands for the 2,000 pounds in each ton of ore. The constant 0.95 stands for the 95 per cent of the copper content, or the net copper that the smelter actually pays for. The dry ton of ore figured in these calculations is the ore with the weight of its moisture content deducted. The moisture content in the ore averages about 10 per cent.

About eight months elapse from the time the ore is mined until the copper is sold. During this period capital is tied up in the metal. Interest on this capital is allowed at the rate of 5 per cent for the eight months. This interest factor is introduced into the equation by multiplying B, the value of copper, by $\frac{100}{105}$, a fraction that represents the lessened value of money by reason of the interest deduction.

In the expression $AC \times 2000 \times 0.95$, used as denominator in the first member of equation *1*, 2,000 pounds of ore multiplied by A, the percentage of copper contained in the ore expressed decimally, multiplied by C, the percentage of copper that is saved by the mill, multiplied by 0.95, the percentage of the copper from the mill that is paid for by the smelter, gives the net amount of copper in pounds recovered from each ton of ore. The expression $\frac{M+F_0+m}{AC \times 2000 \times 0.95}$, means simply the net number of pounds of copper recovered, divided into the cost of recovering it. In the same way, $\frac{f_0+S}{2000D \times 0.95}$ means the net copper recovered from the concentrates at the smelter divided into the cost of recovering it. The whole equation then represents the total net cost of copper with the different factors expressed as mathematical quantities, equated to $B\frac{100}{105}$, which is the value of the copper lessened by an interest charge of 5 per cent. As the equation stands, nothing is charged for the interest lost on the deferred profits. In general, application of the formula interest on the capital tied up in the enterprise should be charged.

By substituting in equation *5* the different costs selected as being well within the factor of safety for the first estimate of the ore values, and simplifying, we get:

$$(6) \qquad A=\frac{1.157}{1235\ (0.1238-0.0157-0.0133)}=\frac{1.157}{1235\ (0.0948)}=\frac{1.157}{117.078}$$

$$=0.00988, \text{ or } 0.988 \text{ per cent.}$$

Now by eliminating M, the cost of mining, from equation *5* and then solving for A, we find what low-grade material, which had to be removed in order to get at richer ore, could profitably be sent to the mill rather than to the waste dump. The figures follow.

$$(7) \qquad A=\frac{0.6887}{117.0533}=0.005882, \text{ or } 0.5882 \text{ per cent.}$$

Equation *7* is equation *6* with the M values—the cost of mining—eliminated. Some rock that contains between 0.5 and 0.8 per cent of copper has to be moved to get at good ore; the cost of sending it to the dump is about the same as that of sending it to the mill. The question to be solved under such conditions is: Having this material mined and loaded because of the necessity of getting it out of the way, what content of copper will decide whether it shall be sent to the mill or to the waste dump. Equation *7* gives the answer—all rock that contains more than 0.5882 per cent of copper should, under the conditions given, go to the mill, and all rock that contains a lesser proportion of copper should go to the waste dump.

FORMULA TO DETERMINE WHETHER ORE SHOULD BE SMELTED IN THE CRUDE, OR MILLED AND THE CONCENTRATES SMELTED.

The formula used to determine what grade of ore from the mine should be shipped direct to the smelter, or to show where the cleavage between smelting ore and concentrating ore should be, is discussed below. When the total cost per pound of copper paid for is the same, whether the ore be smelted in the crude or milled and the concentrates smelted, then the following equation holds:

$$(8)\qquad \frac{M+F_s+S_0}{Ay}+R+E+c=\frac{M+F_0+m}{ACy}+\frac{f_0+S_c}{Dy}+R+E+c.$$

The values substituted in this formula are in general fairly representative of the costs at Santa Rita up to the middle of 1914. Since then improvements have been made.

Values substituted for factors in equation 8.

Factor.	Definition of factor.	Value.
M	=Mining cost per dry ton of ore	\$0.4683
F_s	=Freight on crude ore from mine to smelter per dry ton	1.1111
F_0	=Freight on ore from mine to mill per dry ton	.1111
m	=Milling costs per dry ton of ore	.5776
f_0	=Freight on concentrates, mill to smelter, per dry ton	1.1191
S_0	=Smelting charges for crude ore per dry ton	7.25
S_c	=Smelting charge per dry ton of concentrates	6.352
R	=Refining and delivering per pound of copper paid for	.0106
E	=Export freight, insurance, etc., per pound of copper paid for	.0014
c	=Commission	$\frac{1}{100}B$
B	=Value per pound of copper.	
C	=Percentage of extraction by mill.	
D	=Percentage of copper in the concentrates.	
A	=Percentage of copper per ton of ore (expressed decimally).	
y	=(2000 multiplied by 0.95)=1900.	
0.95	=Percentage of the copper content which is recovered by the smelter (expressed decimally).	

Multiplying equation *8* by Ay we get:

$$(9)\qquad M+F_s+S_o+Ay(R+E+c)=\frac{M+F_o+m}{C}+\frac{A(f_o+S_o)}{D}+Ay(R+E+c)$$

Transposing:

$$(10)\qquad \frac{A(f_o+S_o)}{D}+Ay(R+E+c)-Ay(R+E+c)=M+F_s+S_o-\left(\frac{M+F_o+m}{C}\right)$$

Hence:

$$(11)\qquad A=\frac{\dfrac{M+F_s+S_o}{1}-\dfrac{(M+F_o+m)}{C}}{\dfrac{f_o+S_o}{D}}=\frac{D[C(M+F_s+S_o)-(M+F_o+m)]}{C(f_o+S_o)}$$

If the value of the mill recovery (C) and the percentage of copper the concentrates contain (D) are known, these values can be substituted in equation *11* and the value of A obtained. Below are a few values of A for given values of C and D.

Values of A for given values of C and D.

C	D	A	C	D	A	C	D	A
P. ct.	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*
60	20	18.47	60	25	23.09	60	30	27.71
65	20	18.87	65	25	23.59	65	30	28.31
70	20	19.21	70	25	24.01	70	30	28.82

In other words, with C at 60 per cent and D at 20 per cent, the rock would have to contain 18.47 per cent of copper before the cost of recovering that copper would be the same whether the ore was smelted in the crude or milled and the concentrates smelted. For given values of C and D, the corresponding values for A represent critical values below which the ore should be milled and the concentrates smelted and above which it should be shipped direct to the smelter.

When the profits on every dry ton of ore are equal whether the ore is smelted in the crude or milled and the concentrates smelted, then the following is true: The value of the copper recovered from each dry ton of ore by direct smelting (VCu_s) minus the total cost of recovering it (C_s) is equal to the value of the copper recovered from each dry ton of ore by milling and smelting the concentrates (VCu_{m+s}) minus the cost of all the expenses of treating and handling each ton of ore (C_{m+s}). Putting this in equational form:

(*a*) $VCu_s-C_s=P_s=$ profits from direct smelting.

(*b*) $VCu_{m+s}-C_{m+s}=P_{m+s}=$ profits from milling and smelting the concentrates.

But if $P_s=P_{m+s}$, then

(*c*) $VCu_s-C_s=VCu_{m+s}-C_{m+s}$.

Now, substituting in equation *c* the mathematical expressions for the value of the net copper extracted from each ton treated and the expressions for the cost of treating by both methods, selected from equation *9*, we get:

$$(d)\quad \begin{gathered} \mathrm{ABy-[(M+F_s+S_o)+Ay(R+E+c)]=} \\ \mathrm{ABCy-\left[(M+F_o+m)+\frac{(f_o+S_o)}{D}AC+(R+E+c)ACy\right]} \end{gathered}$$

By substitution of the more fixed costs, etc., we get:

$$(e)\quad 1900\mathrm{AB}-8.8294-22.8\mathrm{A}-19\mathrm{AB}=1900\mathrm{ABC}-1.157-\frac{7.4711\mathrm{AC}}{\mathrm{D}}-22.8\mathrm{AC}-19\mathrm{ABC}$$

Simplifying:

$$(f)\quad 1881\mathrm{AB}-22.8\mathrm{A}=1881\mathrm{ABC}+7.6724-\frac{7.4711\mathrm{AC}}{\mathrm{D}}-22.8\mathrm{AC}$$

Factoring and transposing, we get:

$$1881\mathrm{AB}(1-\mathrm{C})=7.6724+22.8\mathrm{A}(1-\mathrm{C})-\frac{7.4711\mathrm{AC}}{\mathrm{D}}$$

Transposing, we get:

$$1881\mathrm{AB}(1-\mathrm{C})-22.8\mathrm{A}(1-\mathrm{C})=7.6724-\frac{7.4711\mathrm{AC}}{\mathrm{D}}$$

Multiplying through by $-\frac{1}{\mathrm{A}}$ and transposing, we have:

$$\frac{7.6724}{\mathrm{A}}=1881\mathrm{B}(1-\mathrm{C})-22.8(1-\mathrm{C})+\frac{7.4711\mathrm{C}}{\mathrm{D}}$$

Factoring and solving for A, we get:

$$\frac{7.6724}{\mathrm{A}}=(1881\mathrm{B}-22.8)(1-\mathrm{C})+\frac{7.4711\mathrm{C}}{\mathrm{D}}$$

Therefore:

$$(g)\quad \mathrm{A}=\frac{7.6724}{(1881\mathrm{B}-22.8)(1-\mathrm{C})+\frac{7.4711\mathrm{C}}{\mathrm{D}}}$$

Now, by substituting values for B, C, and D, we can solve for A. The value of A thus obtained will be the critical grade or percentage of copper per dry ton of ore, at which the profits will be equal whether the ore be smelted in the crude or milled and the concentrates smelted. If we let D = 20 per cent, C = 60 per cent, and B = 12 cents, we can substitute these values in formula *g* and solve for A, thus:

$$(h)\quad \mathrm{A}=\frac{7.6724}{(1881\times0.12-22.8)(1-0.60)+\frac{7.4711\times0.60}{0.20}}=0.0741, \text{ or } 7.41 \text{ per cent.}$$

If the copper content be greater than 7.41 per cent under the conditions given, the ore should be sent direct to the smelter; if less, it should be sent to the mill.

From the formula represented by equation *h* a table may be made showing the values of A for several values of B, C, and D, thus·

Values of A for several values of B, C, and D, in equation h.

D=20 per cent; C=60 per cent.		D=20 per cent; C=65 per cent.		D=20 per cent; C=70 per cent.	
B	A	B	A	B	A
12 cents	7.41 per cent	12 cents	8.05 per cent	12 cents	8.82 per cent
14 cents	6.47 per cent	14 cents	7.07 per cent	14 cents	7.80 per cent

By giving other values to D, as well as by giving B and C a large range, the table can be made much more extensive. In all cases the value of A thus obtained is the approximate grade of ore at which profits, per dry tone of ore, equalize, whether the treatment is direct smelting or milling and smelting the concentrates.

The expression $\frac{M+F_s+S_o}{Ay}+R+E+c$, taken from formula *8* (p. 30), represents the cost per pound of the copper sold when the ore is treated by direct smelting. By substituting values as given on page 30, simplifying, and rearranging, we get:

$$\frac{0.004647}{A}+0.012+\frac{B}{100}=0.012+\frac{0.004647}{A}+\frac{B}{100}=\text{cost per pound of copper sold when the ore is smelted direct.}$$

Now, by substituting values for A and B, and solving, we get the cost of copper per pound. Subtracting this figure from the selling price gives the profit per pound. If considerable range be given to the values of A and B, a table may be made somewhat as follows:

Cost and profit per pound of copper paid for when ore is smelted in the crude.

Price of copper per pound.		Per cent of copper in the ore.						
		4	5	6	7	8	9	10
Cents.								
12	Cost	$0.129	$0.1061	$0.0907	$0.0796	$0.0713	$0.0648	$0.0597
	Profit	[a].009	.0139	.0293	.0404	.0487	.0552	.0603
14	Cost	.1296	.1063	.0909	.0798	.0715	.0650	.0599
	Profit	.0104	.0337	.0491	.0602	.0685	.0750	.0801
16	Cost	.1298	.1065	.0911	.0800	.0717	0652	.0601
	Profit	.0302	.0535	.0689	.0800	.0883	.0948	.0999
18	Cost	.1300	.1067	.0913	.0801	.0719	.0654	.0603
	Profit	.0500	.0733	.0887	.0999	.1081	.1146	.1197
20	Cost	.1302	.1069	.0915	.0803	.0721	.0656	.0605
	Profit	.0698	.0931	.1085	.1197	.1279	.1344	.1395

[a] Loss of 9 mills per pound of copper.

By substituting 0.05 for A, or 5 per cent, and 0.12 for B, or 12 cents per pound, and solving, we get $0.1061 as the cost per pound of copper by direct smelting of 5 per cent ore, as shown in the above table; subtracting this value from the price of copper, $0.12, we get $0.0139 as the profit per pound. An extensive table may be compiled in this way.

In like manner, from formula *d* (p. 32) we get the expression $[M+F_s+S_o+Ay(R+E+c)]$, which stands for the total cost of mining and treating each dry ton of ore when the process is direct smelting. By substitution of the values given on page 30 the expression reduces to $8.8294+22.8A+19AB$, the cost per dry ton of ore treated. Substituting values and solving gives the cost per ton. By subtracting the figure for cost from the product of the expression $2000\times0.95\times0.05\times0.12$, which represents the net pounds of copper recovered from each ton of 5 per cent ore smelted, multiplied by its selling price, we get the profit per ton treated. A table as extensive as may be necessary can be made thus:

Cost and profit per dry ton (2,000 pounds) of ore smelted in the crude.

Price of copper per pound.		Per cent of copper in the ore.						
		4	5	6	7	8	9	10
Cents.								
12	Cost	$9.833	$10.083	$10.334	$10.585	$10.836	$11.087	$11.337
	Profit	[a].713	1.317	3.346	5.375	7.402	9.433	11.463
14	Cost	9.848	10.102	10.357	10.612	10.866	11.121	11.375
	Profit	.792	3.198	5.603	8.008	10.414	12.819	15.225
16	Cost	9.863	10.121	10.380	10.638	10.897	11.155	11.413
	Profit	2.297	5.079	7.860	10.642	13.425	16.205	18.987
18	Cost	9.878	10.140	10.403	10.665	10.927	11.189	11.451
	Profit	3.802	6.960	10.117	13.264	16.438	19.591	22.750
20	Cost	9.893	10.159	10.426	10.692	10.957	11.223	11.489
	Profit	5.307	8.841	12.374	15.897	19.450	22.977	26.512

[a] Represents a loss of 71.3 cents per ton of ore treated.

By giving a larger range of values to A and B, this table can be greatly extended.

From formula *8* (p. 30) we get the expression $\frac{(M+F_o+m)}{ACy}+\frac{(f_o+S_o)}{Dy}+(R+E+c)$, which represents the cost per pound of the copper actually marketed where the ore is milled and the concentrates smelted. Now, substituting cost values given on page 30 in this expression we get $\frac{1.157}{1900\ AC}+\frac{7.4711}{1900\ D}+0.012+\frac{B}{100}$ = cost per pound of copper sold when the treatment is milling and smelting the concentrates. By giving A, B, C, and D different values a whole series of tables may be compiled showing the cost and the profit per pound of copper sold.

In like manner, from formula *d* (p. 32), the expression $[M+F_o+m+\left(\frac{f_o+S_o}{D}\right)AC+(R+E+c)ACy]$ represents the cost of treating each dry ton of ore by milling and smelting the concentrates. Substituting costs gives $1.157+\frac{7.4711AC}{D}+22.8AC+19ABC$. By substituting a range of values for A, B, C, and D an extensive series of tables can be compiled showing the cost and profit on each dry ton of ore by this method of treatment.

SAMPLING AND ASSAYING METHODS.

PROSPECT-HOLE SAMPLES.

In the development of "ore in sight" by drilling, samples were taken about every 5 feet of hole drilled in the relatively barren material near the surface; but in all other material they were taken every 3 feet. The mixture of water and drill-hole cuttings that composed each sample was run through what was, in effect, a Jones sampler, and quartered down so that sample and washings constituted about a gallon. This ultimate sample was evaporated to dryness on a specially arranged home-made furnace, consisting of a fire box and a heating chamber built from $\frac{1}{8}$-inch boiler plate, somewhat as shown in figure 4.

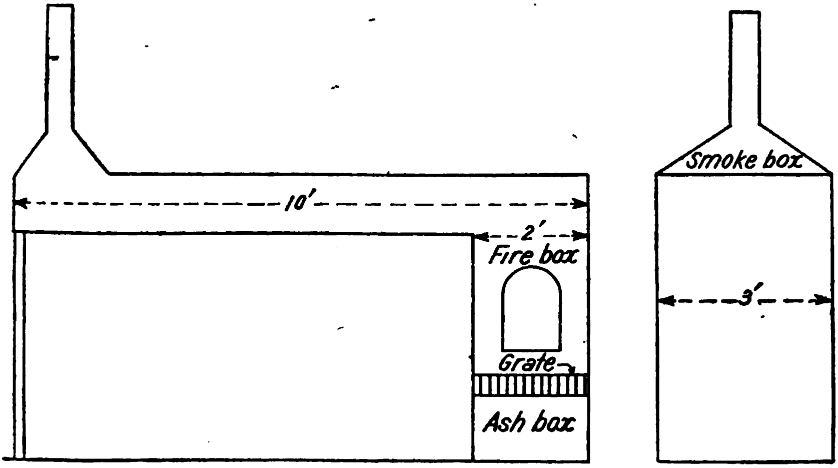


FIGURE 4.—Side and end views of improvised furnace for evaporating drill samples of ore. Can be made by blacksmith out of $\frac{3}{16}$-inch or $\frac{1}{4}$-inch boiler plate. Smoke box of heavy sheet iron; smokestack, 6 feet of ordinary 6-inch stovepipe; evaporating pans, about 18 by 18 inches and 6 inches high, made of heavy sheet iron.

Some of the rejected part of each sample was carefully panned and the concentrates examined to get information regarding the character of the rock through which the hole was passing. When notes covering these data had been made, the panned material was put into paper envelopes and filed away.

The dried sample was tagged with the number of the hole and the depth at which it was taken, and sent to the sample room. There, if necessary, it was run through a small gyratory crusher, the largest pieces from which were about one-fourth of an inch in diameter; then after careful rolling on a mixing cloth it was reduced on a Jones sampler to about 150 or 200 grams. After that came grinding in a Braun disk pulverizer, followed by more rolling on a mixing sheet. Screening through a 150-mesh to 200-mesh screen removed any metallic copper, and this, if present, was weighed and figured into the ore values as 90 per cent pure. The pulp was then sent to the assay shop, where the determination was made, mostly by Lowe's potassium permanganate method. Each assay was given a serial number in addition to the number of the hole and the depth below the surface from which it had been taken, and the part of the pulp not required in the assay was put into a paper envelope and carefully filed away.

GOLD ASSAYS.

Since the summer of 1914 a large number of gold assays have been made. As a rule, the amount of gold present is small, so that charges of 4 assay tons[a] have had to be used to get a gold bead of a size that might be weighed conveniently. The smelter pays for the gold in the concentrates when its value is 60 cents or more per ton. Assuming a concentration of 10 into 1, and a saving of 70 per cent in the mill, a gold content of only 9 cents per ton in the ore would give a revenue from the smelter of 63 cents per ton of concentrates. Obviously the presence of even small amounts of gold favors the practice of making the concentrates as clean as possible, and during the winter of 1914–15 important progress was made in this direction at the Hurley mill.

BLAST-HOLE AND PIT SAMPLES.

Where more detailed information is necessary as mining proceeds, samples are taken from the blast holes. Such sampling shows with considerable exactness the value of the rock in place ahead of the steam shovels. Samples are taken—a small shovelful here and there—from the broken rock in front of each steam shovel. These are called pit samples and may be gathered every hour or so. Rough determinations, by the colorimetric method, are made on these, as they are only for the purpose of showing whether the rock is running about as it has been indicated by the cross sections and the estimates. Samples are also taken from every car of ore loaded, and these are checked against other assays of the ore body and against mill returns, tailings assays, etc. Formerly, samples were also taken by carefully cutting across the face of many of the benches with a small pick or a hammer and moil; a ladder was used to get at the upper part of the face. Few bench samples are taken now. Moisture determinations were made at various times on the ore and rock.

STEAM-SHOVEL SAMPLES.

Steam-shovel samples are occasionally taken and consist of a "grab" shovelful from each steam-shovel dipperful after it has been put on the car.

---

[a] An assay ton contains 29.166 grams.

To summarize, all four kinds of samples are currently taken at Santa Rita, as follows:

1. Prospect-hole samples.
2. Pit samples.
3. Car and steam-shovel samples.
4. Blast-hole samples.

Moisture determinations are made on many of the samples. On the average, about 2,400 assays and determinations are made per month. The assay staff consists of an assayer, one assistant, and two men in the sample shop. The latter attend to the grinding of the samples, preparing the pulp, etc.

SAMPLE SHOP AND ASSAY SHOP.

The sample shop, a one-story structure, having a floor space of about 500 square feet, is fitted with the following appliances: 1 Blake jaw crusher, with opening 10 by 5 inches, crushing to 2 inches, to which a revolving sampler is attached; a small Blake crusher, with an opening 3 by 4 inches, crushing to ¼ inch; a gyratory crusher giving a maximum size of ¼ inch; a Braun disk pulverizer; a gasoline stove for drying, and many other accessories. Power is furnished by an Allis-Chalmers 7.5 horsepower motor.

The assay shop, with almost 1,000 square feet of floor space, contains a balance room, a laboratory for wet assays, and a furnace room. In the latter are a smelting and a cupelling furnace, each fired by gasoline, vaporized under about 50 pounds of air pressure, and blown in through Cary hydrocarbon burners.

NUMBER AND COST OF ASSAYS.

The following table gives some idea of the number of assays made per month and the cost of making them:

Cost of sampling and assaying at Santa Rita.

Month.	Number of assays.	Cost per assay.	Mining costs.			Stripping costs.		
			Labor.	Supplies.	Cost per ton.	Labor.	Supplies.	Cost per cubic yd.
1914.								
January	3,610	$0.46	$610.73	$106.69	$0.00366	$802.72	$128.39	$0.00302
February	2,159	.80	575.72	112.69	.00365	882.56	149.88	.00365
March	3,001	.66	623.36	151.46	.00349	997.96	193.41	.00345
August	[a] 1,335	.86	370.84	182.67	.00454	408.74	182.38	.00317
September	[b] 1,475	.42	207.90	58.03	.00255	276.14	78.74	.00223

[a] 66 of these assays were for gold.
[b] 130 of these assays were for gold.

As is indicated by the table, the figures cover ore and waste. The ore assay costs are charged against mining, and the costs of assaying waste are charged against stripping. The cost of the labor for assaying and sampling and the cost of the supplies used in connection with the assays are kept separate, as they are in all the other accounts.

The assay cost is distributed per ton according to the tons of ore mined, and per cubic yard according to the cubic yards of waste moved, as shown by the table.

The permanganate method is used for prospect drill holes and for car samples. The colorimetric method is used for pit and shovel samples where absolute accuracy is not considered necessary. The determination of iron in many samples is made as it is indicative of the degree to which the ore will concentrate.

The wide variation in the cost of the assays is caused by the wide variation in the amount of crushing and mixing to be done on them—whether permanganate or colorimetric determinations or fire assays are made; the relative number of each, and the total number of all assays for the month; the amount and cost of the chemicals used, etc.

FORMS USED IN ASSAY SHOP.

Forms 6 to 9 following are used in the assay shop and are convenient aids to bookkeeping.

FORM 6.—*Form of general assay certificate issued by assayer.*[a]

ASSAY CERTIFICATE.

Per ton of 2,000 lbs. SANTA RITA, N. MEX., ..., 191...

Sample No.	Date.	S. S. No.	Ore body.	Bench.	Description.	% Silica.	% Sulphur.	% Iron.	% Copper.	Oz. gold.	Oz. silver.			

...., *Assayer.*

---

[a] Size of form as used by operating company, 5½ by 7½ inches.

Form 7.—*Form used by assayer to report on steam-shovel samples.*[a]

STEAM-SHOVEL SAMPLES—LABORATORY REPORT.

Sample No.	S. S. No.	Date.	Shift.	Cars waste.	Cars ore.	Wt. of met.	Wt. of pulp.	% Met. Cu.	% Perm. Cu.	% Cu. total.	

[a] Samples consisted of a small shovelful taken from each steam shovelful that went onto a car. Samples of this kind are not now usually taken. Form used by operating company is 6 inches wide by 9 inches long.

Form 8.—*Form for presenting comparative data regarding steam-shovel samples.*[a]

COMPARATIVE STATEMENT—STEAM-SHOVEL SAMPLES.

Day Shift....................., 191..

Steam Shovel No...................

.... Ore body.

.... Bench.

Time.	Pit sample % Cu.	Mined as—		Bench sample—		Mined as—	
		Ore.	Waste.	No.	% Cu.	Ore.	Waste.

Average % Cu.

Car sample % Cu.

Car sample % Cu. Ore shipped to mill.

SPECIAL PIT SAMPLES.

Time.	Taken from.	Ore % Cu.	Waste % Cu.

Remarks:

---

[a] Only one form is shown; the record used by the operating company has five of these forms (3¾ by 8¾ inches) on a sheet, the sheet with margins being 10 by 20½ inches. Each sheet, bound through two holes in the left margin, is kept in a heavy binder.

FORM 9.—*Form used in making daily report on mine samples.*[a]

DAILY REPORT OF MINE SAMPLES.

....19....

Mine sample number.	Description.			Per cent copper.			Remarks.
	Mine.	Level.	Working.	Pulp.	Met.	Total.	

....

---

[a] Size of form used by operating company, 8½ inches wide by 10¾ inches long.

METHOD OF SAMPLING OLD DUMPS.

Sampling on the present property, on an extensive scale, was first begun by Mr. John M. Sully, in 1906. In gathering samples from the large dumps left from many thousands of feet of old workings, he used the following method:

The dumps were laid off into 25-foot squares, with levels run to the corners of each square, to determine its elevation. The contour of the ground on which the dump rested was estimated as nearly as possible, from the general surface configuration of the land. From the surface elevation and the probable elevation of the bottom of the dump, the yardage in each square prism was estimated. Each square, as laid off on the dump surface, was given a number, and about 4 holes were dug into it, as deep as could be conveniently made with a long-handled shovel. Every second shovelful was retained; the sample thus gathered was rolled and quartered on a canvas and put into a tagged canvas sack. Mr. Sully gathered 1,143 samples from the old dumps, and a total of 8,190 from the dumps and the old workings.

DETAILS OF MINING IN SANTA RITA DISTRICT.

GENERAL ORGANIZATION OF OPERATING COMPANY.

In opening an open-cut mine on an extensive scale, the initial cost is heavy. The equipment necessary is expensive, including steam shovels, locomotives, and cars, and railroad track and a water-supply system must be constructed, to say nothing of the concentrating plant to handle vast tonnages of ore, the power plant to furnish the motive force, and the machine and other shops to keep the whole vast plant in repair.

Any large and successful organization is developed slowly. The factor that wins in business or in sport is practically always well-organized "team work," or efficiency of production. If a mistake occurs the organization must be such that the responsibility for the mistake can be traced to its source at once, and the right remedy be quickly applied in the right place. The management of the Chino company has been successful in building up an efficient and loyal organization, one illustrating the benefits that may be derived from the application of science and of business methods to the solution of mining problems. The company's organization chart, or "flow sheet" of responsibility, as worked out by Mr. L. E. Foster, the assistant superintendent, is shown in Plate IV.

MINING AND ESTIMATION OF ORE BY BENCHES.

In stripping and mining, each steam shovel took off a slice of ground about 25 feet wide, and as the work progressed terraces or benches 100 feet or more wide were formed around the sides of the great open cuts (Pl. V, *A*). It was found that where the bench was less than 30 feet high, the material could not be excavated as economically as where it was, say, 50 feet or more high.

There were three chief factors that regulated the height of each bench: (1) If a bank stood fairly steep for some time and then caved, it was found best to have the bench not more than 30 feet high. The caving of 30-foot banks caused little delay or damage to the shovels working below them, whereas the caving of higher banks often caused serious trouble. Where a bank caved slowly and kept gradually coming to the shovel at about the same general slope, it could be worked in benches of 100 feet or more if necessary. (2) If the waste material on top of the ore was, say, only 15 feet thick, then, of course, the bench at that place was only 15 feet high. On the other hand, if the overburden was only, say, 1 or 2 feet thick, it was probably found cheaper to let it go in with the ore without stripping. (3) Where the drill holes indicated considerable relatively barren rock between upper and lower ore masses, then, as far as possible, the benches were planned so that the top ore was taken off the waste rock in one cut or bench, and the waste rock off the lower ore in the second bench, and the lower ore was taken out clean in a third bench. If the ore bodies and waste mass were thick enough, they were generally taken out in two or more separate benches. Barren areas of considerable size were also considered in planning the benches.

In other words, benches and tracks were planned (see Pl. III) to conform, as nearly as practicable, to the shape of the ore body, so that ore could be separated from waste and mined as economically as possible. In general, however, it was found best to have the benches about 50 feet apart; that is, one bench about 50 feet above the other.

The different classes of ore and the waste were calculated for each level, or bench, in order to have convenient information as to just what production should be expected from each.

Tables were also made to show the ore tonnage by benches; that is, the tonnage of the first bench plus the tonnage of the third bench, etc. Such tables permit convenient comparison of what has been taken out from the different benches plus the material calculated to remain with the total ore for each bench, etc. These bench calculations were made from the same data and by the same general method as the triangular-prism estimates. Summing up the ore classes, calculated by benches, served as a sort of check on the other calculations of total ore.

BLAST-HOLE DRILLING.

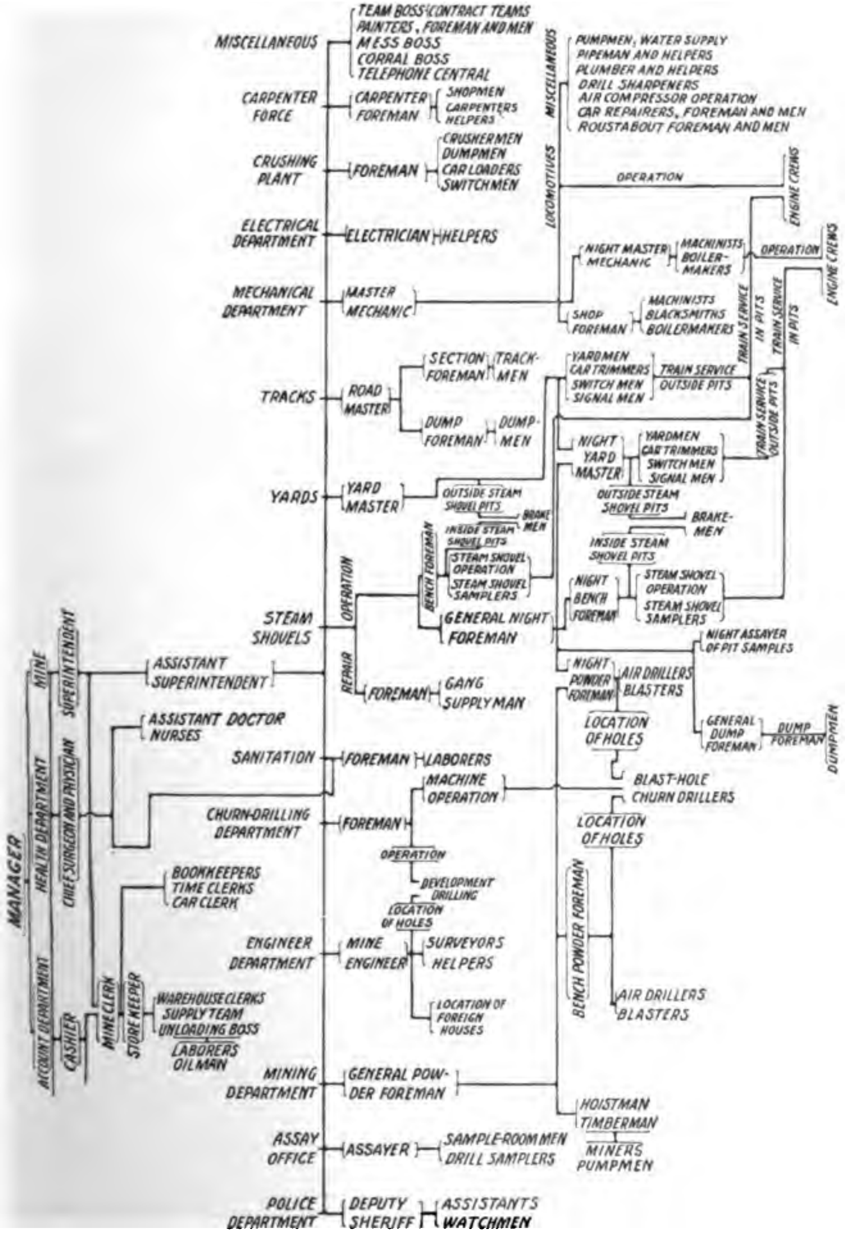
The blast-hole drilling at Santa Rita aggregates several linear miles per month. Most of it is done by churn drills. However, toe holes at the foot of slopes are locally necessary, and these, together with the shallow, levelling-off bottom holes occasionally needed in front of steam shovels, are put down with compressed-air tripod drills. Small hammer drills have been used, to some extent, for drilling short holes to blast bowlders too large for the steam shovels to handle. However, their use has been practically discontinued, because it is generally cheaper to use more powder and to "dobe" or "bulldoze" the rock than to keep a shovel idle while it is being drilled.

TYPES OF CHURN DRILLS USED.

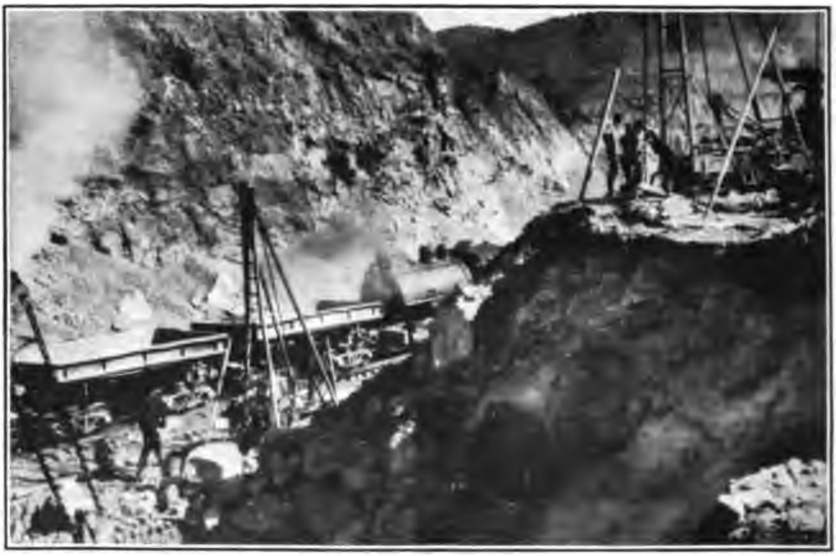
The types of churn drill mostly used at Santa Rita are the No. 12 and the No. 14 Cyclone drills for big blast holes. They are fitted with Cook 12-horsepower vertical gasoline motors, have traction gearing, and use 4-inch to 8-inch bits. Gas-engine oil, instead of gasoline, is used, and the consumption averages about 6½ gallons per 10-hour shift. The speed of drilling varies widely with variation in the hardness of the rock; however, the general average for blast holes not more than 65 feet deep is said to be about 20 to 30 feet per 10-hour shift. The crew of each machine consists of a driller and a helper; these two men can move the drill, even over rough ground, by turning on the traction gearing.

Each drill is fitted with equipment that enables the driller to sharpen his own bits. In general, the rock is soft enough so that one to two sharpenings per shift suffice. The blast holes are sampled if special information from particular localities is needed.

A few of the older steam-driven churn drills are now operated by air, piped from the compressor. In general, they are not as satisfactory as the oil-motor machines. There have been as many as 18 churn drills, both prospect and blast-hole, at work on the property at the same time.

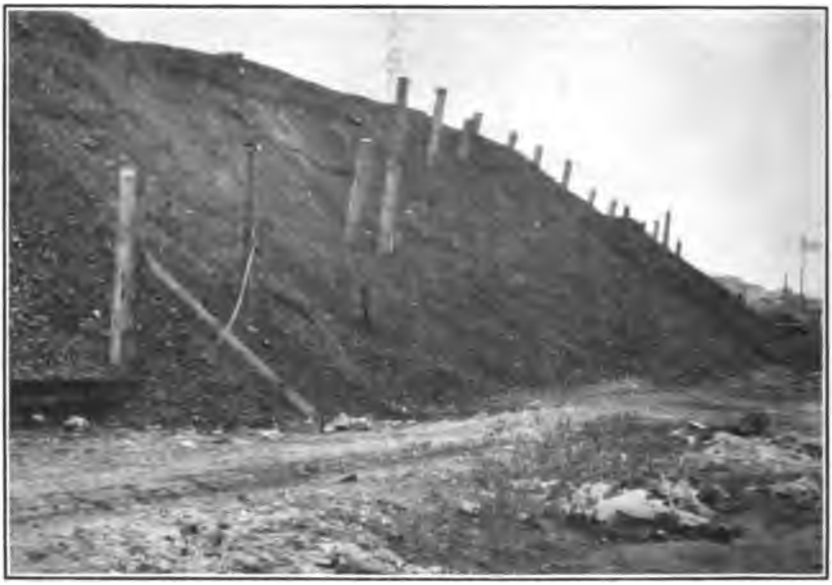


ORGANIZATION CHART OF THE CHINO COPPER CO.



A. VIEW OF A BENCH OR TERRACE OF ORE, SHOWING METHOD OF MINING.

On the right, drills are at work on a bench just ahead of a steam shovel. The chain of the shovel can be seen at the left. A train of cars is on the loading track below the bench that is being drilled.



B. PILE OF COAL CONTAINING WOODEN VENTILATING STACKS TO PREVENT SPONTANEOUS COMBUSTION.

The hydrothermally altered and considerably sheared character of the ore-containing rock facilitates the use of churn drills and is favorable for low drilling costs. Little trouble is encountered in putting down blast holes. Even in cutting across sloping sheared zones, a little care generally guards against much deviation from the vertical.

OPERATION AND EFFICIENCY OF BLAST-HOLE CHURN DRILLS.

Cyclone churn drills, models Nos. 12 and 14 for deep blast holes, are used to put down blast holes. Records of the performance of these are kept and constitute valuable information on the efficiency of churn drilling in general. Records for two typical months, January and August, 1914, are given below:

Record of blast-hole churn drilling for January, 1914.

DAY SHIFT.

Drill No.	Feet drilled.	Total working hours.	Average feet per hour.	Time spent in drilling.	Time delayed.	Time spent in repairing.	Average feet per hour of drilling.
				Per cent.	*Per cent.*	*Per cent.*	
1	630	280	2.25	72.8	19.3	7.9	3.09
2	876	310	2.83	61.9	25.2	12.9	4.56
10	1,235	327	3.78	84.1	11.9	4.0	4.49
11	974	310	3.14	75.2	16.1	8.7	4.13
12	1,212	310	3.91	86.8	8.4	4.8	4.51
13	941	322	2.92	73.9	15.8	10.3	3.95
14	924	311	2.97	78.5	17.0	4.5	3.79
15	952	310	3.07	79.0	20.0	1.0	3.88
NIGHT SHIFT.							
1	670	260	2.58	86.5	13.1	0.4	2.98
2	1,144	298	3.84	84.2	13.8	2.0	4.56
10	1,272	321	3.95	88.2	7.8	4.0	4.50
11	928	266	3.49	89.9	8.6	1.5	3.88
12	1,244	290	4.29	98.0	1.7	.3	4.38
13	1,000	329	3.04	83.6	15.8	.6	3.64
14	953	275	3.47	90.5	9.5	....	3.83
15	1,151	310	3.71	90.3	9.4	.3	4.12
TOTAL.							
1	1,300	540	2.41	79.4	16.3	4.3	3.03
2	2,020	608	3.32	72.8	19.6	7.6	4.56
10	2,507	648	3.87	86.1	9.9	4.0	4.49
11	1,902	576	3.30	81.9	12.7	5.4	4.03
12	2,456	600	4.09	92.2	5.1	2.7	4.44
13	1,941	651	2.98	78.8	15.8	5.4	3.78
14	1,877	586	3.20	84.1	13.5	2.4	3.81
15	2,103	620	3.39	84.7	14.7	.6	4.01
Total for month	16,106	4,829	3.34	82.5	13.4	4.1	4.04

Record of blast-hole churn drilling for August, 1914.

DAY SHIFT.

Drill No.	Feet drilled.	Total working hours.	Average feet per hour.	Time spent in drilling.	Time delayed.	Time spent in repairing.	Average feet per hour of drilling.
				Per cent.	*Per cent.*	*Per cent.*	
1	725	186	3.90	95.2	4.8	....	4.09
2	1,073	235	4.57	95.3	4.7	....	4.79
10	1,049	244	4.30	91.8	2.0	6.2	4.68
11	1,082	256	4.23	92.6	4.7	2.7	4.56
12	788	187	4.22	89.8	7.0	3.2	4.69
13	814	232	3.51	83.2	2.1	14.7	4.22
14	383	185	2.07	75.1	18.4	6.5	2.75
15	963	246	3.91	86.6	8.5	4.9	4.52
16	988	246	4.02	94.3	2.0	3.7	4.26
NIGHT SHIFT.							
1	....	....	....	....	....	....	....
2	125	30	4.17	100.0	....	....	4.17
10	477	129	3.76	97.7	0.8	1.5	3.79
11	82	39	2.10	74.3	23.1	2.6	2.83
12	182	44	4.14	91.0	4.5	4.5	4.56
13	304	81	3.75	95.1	4.9	....	3.95
14	75	39	1.92	69.2	28.2	2.6	2.78
15	....	....	....	....	....	....	....
16	352	94	3.74	95.7	4.3	....	3.92
TOTAL.							
1	725	186	3.90	95.2	4.8	....	4.09
2	1,198	265	4.52	95.8	4.2	....	4.72
10	1,526	373	4.09	93.8	1.6	4.6	4.36
11	1,164	295	3.95	90.1	7.2	2.7	4.38
12	970	231	4.20	90.0	6.5	3.5	4.67
13	1,118	313	3.57	86.2	2.9	10.9	4.14
14	458	224	2.04	74.0	20.2	5.8	2.76
15	963	246	3.91	86.6	8.5	4.9	4.52
16	1,340	340	3.94	94.6	2.7	2.7	4.16
Total for month	9,489	2,473	3.84	90.0	5.9	4.1	4.26

COSTS OF BLAST-HOLE CHURN DRILLING.

The cost of drilling blast holes with churn drills are fairly well illustrated by the figures for the representative months of January and August, 1914, as given in the tabulation following:

Record of costs of blast-hole churn drilling.

AUGUST, 1914.

Churn drill No.	Cost of labor.	Cost of supplies.	Total.	Feet drilled.	Cost per foot.
1	$229.43	$73.36	$302.79	725	$0.4176
2	311.52	76.49	388.01	1,198	.3239
Total for steam drills using air	540.95	149.85	690.80	1,923	.3592
10	383.42	58.25	441.67	1,526	.2894
11	317.99	100.68	418.67	1,164	.3597
12	302.49	89.93	392.42	970	.4046
13	361.37	147.90	509.27	1,118	.4555
14	274.08	106.00	380.08	458	.8299
15	261.02	34.17	295.19	963	.3065
16	377.13	70.19	447.32	1,340	.3338
Total for gasoline drills	2,277.50	607.12	2,884.62	7,539	.3826
Total for all churn drills	2,818.45	756.97	3,575.42	9,462	.3779

Record of costs of blast-hole churn drilling—Continued.

JANUARY, 1914.

Churn drill No.	Cost of labor.	Cost of supplies.	Total.	Feet drilled.	Cost per foot.
1	$595.59	$183.40	$778.99	1,300	$0.599
2	637.90	193.08	830.98	2,020	.411
Total for steam drills using air	1,233.49	376.48	1,609.97	3,320	.485
10	668.24	195.21	863.45	2,507	.344
11	594.89	242.66	837.55	1,902	.440
12	622.02	176.92	798.94	2,456	.325
13	653.24	190.99	844.23	1,941	.435
14	607.87	135.12	742.99	1,877	.396
15	618.97	244.88	863.85	2,103	.411
Total for gasoline drills	3,765.23	1,185.78	4,951.01	12,786	.387
Total for all churn drills	4,998.72	1,562.26	6,560.98	16,106	.407

The total cost of drilling per ton of ore mined is about 1 cent; and per cubic yard of material stripped, about 1.3 cents. The reasons for the cost per foot of blast-hole drilling being so much less than the cost of prospect drilling (p. 14) are that the prospect holes were larger and averaged eight to ten times as deep as those for blasting; also the cost of sampling was included in the prospect-hole costs.

TYPES OF TRIPOD DRILLS USED.

Two types of tripod drill, the Sullivan and the Ingersoll-Rand, are used where the rock is hard enough so that toe and shallow bottom holes are necessary. Both these makes, having 3¼-inch and 3⅝-inch cylinders, were satisfactory. They are operated by air under about a 90-pound pressure, piped from the central compressor plant. The steel used is mostly of the 1¼-inch crescent brand. It is sharpened by a No. 3 Leyner drill-sharpening machine. Machine-sharpened drills have given better service at a smaller cost than was obtained from hand-sharpened drills.

COSTS OF DRILLING BLAST HOLES BY AIR DRILLS.

During January, 1914, air-driven tripod drills drilled 9,875 linear feet of holes at a cost of 46½ cents per foot; during August they made 6,951 feet of holes at the rate of 32½ cents per foot. This great difference in rate of cost seems to be due to greater ease of drilling during August.

USE OF HAMMER DRILLS.

Where bowlders, too large to be handled by a steam shovel, remain after a bench blast, and where loading will not be too much impeded by drilling, or "block holing," instead of by "dobe" blasting, the "block holing" is done by small air hammer drills of the Sullivan D. C., 19 type, using 2-inch bits. Air is supplied to them through flexible armored hose from a supply line running along the top of the cuts. The exhaust from the machine passes out through a hole in the bit, thereby automatically cleaning the hole of cuttings. From 1½ to 3 inches of hole can be drilled per minute with this machine, and it can be operated by one man. The operator is paid $3.75 per day. Only one of these drills was in use at the time the mine was visited, because it had been found that "dobe" blasting of bowlders could be done more quickly and interfered less with loading operations, and hence, in the long run, was more economical.

BLASTING.

The object of blasting in metal mines where there are rich brittle sulphides and carbonates is generally, as in coal mines and quarries, to break material loose from the main mass without pulverizing it greatly. On the other hand, if the broken rock is subsequently to be crushed for recovery of its values or for engineering operations the aim then is that the blasting shall break the rock loose and at the same time pulverize it as much as is possible, consistent with the efficient use of explosives. At Santa Rita, of course, the aim in blasting the ore-bearing rock is to pulverize it. Any fragments of broken rock that remain too large to enter a 3-yard steam-shovel dipper have to be broken by secondary blasting at considerable extra cost.

EXPLOSIVES USED.

In breaking the rock in a safe and efficient manner the following kinds of explosives are those most extensively used:

(*a*) Dupont FF and FFF black blasting powder.

(*b*) Dupont quarry powder.

(*c*) Dupont Repauno low-freezing dynamite, 30, 40, and 60 per cent strengths.

(*d*) Trojan powder No. 2 in bags, and 40 per cent strength, in sticks.

Black powder is used for breaking and loosening up soil, hardpan, or soft rock; and quarry powder, instead of the 40 per cent dynamite, is used for soft or fissured rock. Repauno 40 per cent strength low-freezing dynamite is used to spring holes for primers and to blast holes that contain water; it is also used for holes in medium hard rock, and for "tight holes" in broken formations. For "dobe" shots 40 per cent Repauno dynamite is used almost exclusively. Repauno 60 per cent strength dynamite is used only in the hardest rock formations. Trojan powder, packed in 12½-pound waterproof paper bags, four bags to the box, is used most in medium hard or broken rock. This powder is understood by miners to be of equal strength to 40 per cent dynamite, but on account of its loose, granulated form it fills in the cracks and crevices in broken formations, thus excluding air spaces and increasing the blasting efficiency. Trojan sticks are also used for "springing" or chambering wet holes.

Detonation by fuse and detonator is used for springing holes and for "dobe" or "bulldoze" blasting. All the heavy bank-hole blasting and some of the heavy springing charges are detonated with electric detonators. The brands of fuse used are "Sylvanite" and "Cactus." Sylvanite is an asphaltum-covered, triple-wound, cord fuse, which has a burning-rate of 16 inches per minute. Cactus is a triple-wound cord fuse, with a white clay-compound waterproof covering. It has a burning rate of about 20 inches per minute, and on account of its white color is generally used for night work. No. 7 detonators are used with these fuses. Where the detonation is by electric current, No. 7 electric detonators are used.

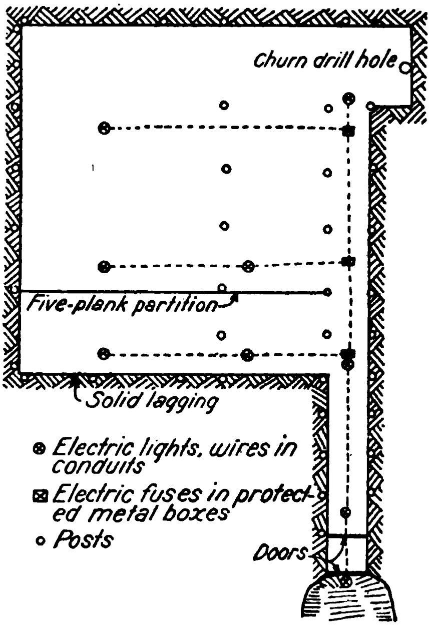

Churn drill hole
Five-plank partition
Solid lagging
⊗ Electric lights, wires in conduits
⊠ Electric fuses in protected metal boxes
○ Posts
Doors

FIGURE 5.—Plan of typical underground explosives magazine in Santa Rita district.

STORAGE AND HANDLING OF EXPLOSIVES.

The main magazines have each a capacity of about 130 tons. Each is formed by a drift into the hillside so that a chamber about 30 by 25 feet, and 8 feet high, is cut into the solid rock (fig. 5). Each chamber is secured by double sets of timbers, with stulls or legs set about 5 feet apart, and with the roof and sides lagged. From the entrance to the back wall a 5-foot passageway is kept clear. The explosives are segregated according to kind and are piled in tiers in the chamber. These underground magazines afford safety against stray bullets, etc., and give what is necessary in the storing of explosives—a comparatively even temperature that remains above freezing during summer and winter. The floor of each of these chambers is covered with several inches of mill tailings. Thus a soft smooth surface, which acts as a cushion to the cases of explosives, is insured.

The magazines have to be kept well ventilated in order that they may not become overheated in summer and that gases from the stored materials may not accumulate in them. To this end, a 6-inch churn-drill hole connects the inner part of the passage in each magazine with the surface (fig. 5). These bore holes are protected on the surface with hooded stacks about 12 feet high, the upper 3 feet of which are perforated.

The entrance to the magazines is guarded by two double-planked, steel-faced, swinging doors, one about 6 feet inside the other. The outside door is perforated in the upper half, and the inside one in the lower half, for the admission of air (Pl. VI, *A*).

From the magazines the explosives are distributed by wagons to several small portable powder stations. There are two of these stations to every steam shovel—one for the high explosives and one for black powder—so they are known as shovel powder houses. They consist of skeleton frames, made of 2½-inch by 2½-inch material, 7 by 4 feet by 4½ feet high, and covered with corrugated sheet iron. Each of these little houses has a capacity of 3,500 pounds of high explosives, or 4,000 pounds of black powder. However, they seldom contain more than a wagon-load—2,000 to 3,000 pounds. Carrying handles, consisting of scantlings 2 by 4 inches in size, are nailed to the sides.

When black powder is needed in large quantities at any point, a sled with a pointed corrugated sheet-iron roof and drawn by a team of horses, is used to transport it from the shovel powder house to the place of use.

Little trouble is experienced from the freezing of explosives, as the operating company generally buys only the low-freezing or nonfreezing brands, and as the magazine temperature is always above freezing. During exceptionally cold weather hot-water thawing chambers are used and then the "powder" has to be carried a considerable distance from the magazines.

"SPRINGING" OR CHAMBERING HOLES.

Before the drill holes are loaded and blasted, one or more small charges are exploded in the bottom of each, to make an enlarged chamber for the final charge. This is known as "springing" or chambering the holes. The aim is to make so large a chamber at the bottom of the hole that the charge may be concentrated into so compact a mass that none of it will fail to detonate, and that the explosive energy may be concentrated where it will give the maximum effect. The springing eliminates any water that may be in the hole, but it generally increases the liability of an inflow of water by shattering and fissuring the surrounding rock. If granulated powder can be forced into these fissured places the effectiveness of the blast is greatly increased.

Where the rock to be broken is relatively soft and weak, and the drill holes not more than, say, 30 feet deep and not over a third of that distance back from the face of a steam-shovel cut, one "springing" is usually all that is necessary. Where the rock is hard and tough, or where the holes are very deep, as many as four successive springing charges may be required before a chamber large enough for

A. ENTRANCE TO UNDERGROUND EXPLOSIVES MAGAZINE.

B. EXPLODING A SPRINGING CHARGE IN A BANK HOLE.

Note how the water and rock shoot out at the surface.

the final blasting charge is made. After each successive "springing" the hole must be cooled off, generally with water, before another charge is introduced.

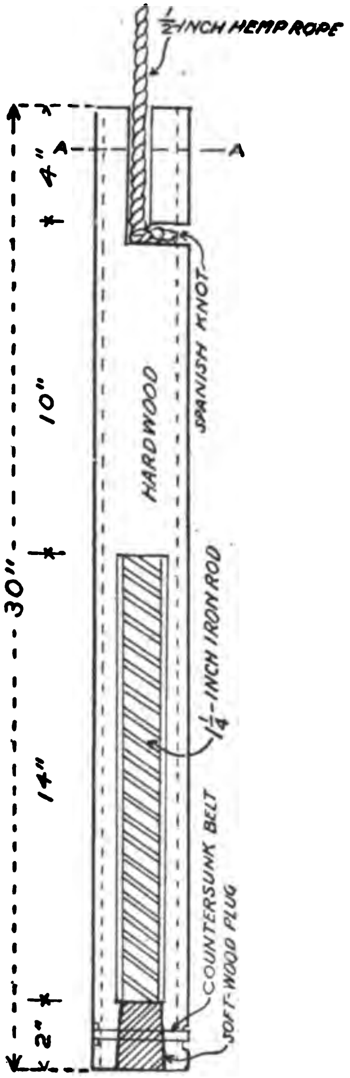
The following are average charges for each successive "springing," in medium-hard rock:

Average charges for "springing" or chambering drill holes.

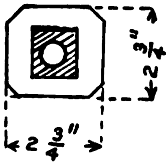
Springing No.	Bank holes.		Toe holes.	
	Number of primers used.	Number of sticks of dynamite used.	Number of primers used.	Number of sticks of dynamite used.
1	1	5	1	2
2	1	11	1	5
3	2	21	1	9
4	2	28	....	....

For "springing" holes 40 per cent strength dynamite or other high explosive, cut into quarter or third sticks, is generally used. It is detonated with a No. 7 detonator crimped onto 24 inches of fuse.

Both before and after "springing" the drill holes are sounded by a specially constructed plummet—a billet of hardwood about 30 inches long and having a metal weight in the lower end. This sounding apparatus, known as a "springing gage" (fig. 6), is attached to a rope and lowered into the hole. By tilting the upper end of it from side to side while the lower end rests on the bottom (fig. 7) the experienced chargeman can estimate the size of the hole. With this gage he also measures the depth of water in the hole. A small hand mirror is at times very useful for reflecting



LONGITUDINAL CROSS SECTION



SECTION AA.

FIGURE 6.—Longitudinal and transverse cross sections of "springing gage."

light into the hole, thus enabling inspection of the upper 10 to 30 feet.

The procedure in "springing" holes is somewhat as follows: The chargeman lowers the springing gage into the hole and notes its size and depth, the depth of water, etc. He then drops in the springing charge, and again lowers the gage to determine whether the charge has reached the bottom. A fuse, passed diagonally through a stick of dynamite about 2 inches from one end, extends along the side of the stick to the other end, into which the end containing the detonator is inserted about 2½ inches (fig. 8, A).[a] This stick of dynamite, with cap and fuse attached, is called a "primer." The fuse is then lighted and the primer is dropped or lowered into the charged hole, followed by a hurried lowering of the gage to show whether the primer has reached the bottom. After this 2 to 4 gallons of water is poured in as "stemming," the gage is quickly withdrawn, and the chargeman retreats to a safe distance. After the detonation, the gage is again lowered and the size of the chamber estimated. If necessary other "springing" charges are used, each about twice as large as the preceding one, until the hole is sufficiently widened or chambered out at the base to receive a proper blasting charge (fig. 9). For the third "springing," where the holes are over 30 feet deep, and often for the second "springing," if much water is present, electric detonation is used (fig. 8, B and C). Where there are several holes to be fired simultaneously electricity is employed instead of fuse.

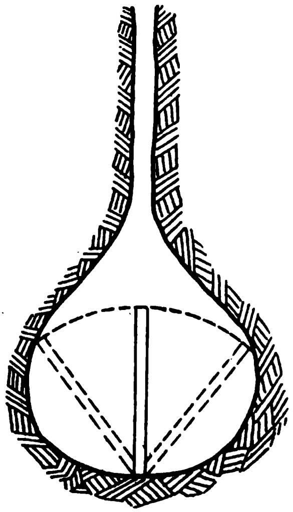


FIGURE 7.—View of chambered drill hole showing how "springing gage" is used.

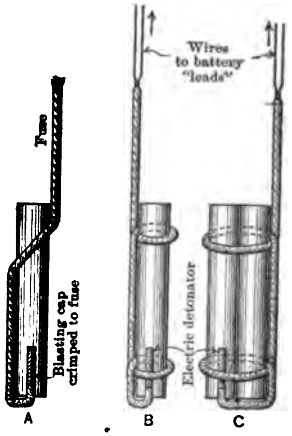


FIGURE 8.—Arrangement of fuses for different primers. A, for detonation with fuse, as practiced in Santa Rita district; B, for electrical detonation, single primer; C, for electrical detonation, double primer.

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[a] The Bureau of Mines does not recommend the lacing of a fuse through a primer.

The lead wires or detonating wires are connected in series and the detonators tested with a galvanometer. The lead wires are then connected with a battery and the charges are fired (Pl. VI, *B*).

CHARGING AND FIRING.

The blasting squad usually consists of a charge man or powder man and four helpers. The charge man places all explosives in the drill holes, except in the "springing" of short holes, when the work may be done by the assistant charge man. The helpers carry the explosives and accessories to the charge man while he is gaging the hole and charging it.

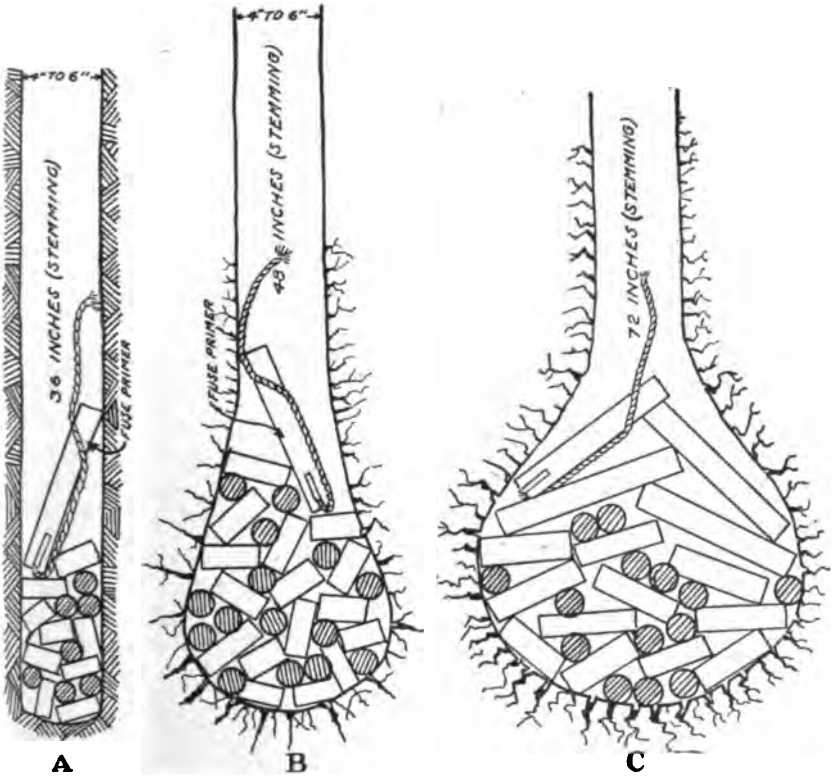

FIGURE 9.—"Springing" charges in bank holes in dry soil or soft rock. Water stemming only. A, charge for first "springing"; B, charge for second "springing"; C, charge for third "springing."

The amount of the charge for each hole varies according to the toughness of the rock, the position of the hole with relation to the face and to other holes, and its depth and extent of chambering. The drill holes to be blasted are "bank holes"—those drilled vertically on each bench and situated back approximately one-third of their depth from the edge of the bench—and "toe holes"—those driven into the face of the bench, say, 4 to 6 feet above the shovel level at the foot of the bench. The toe holes are driven with a downward slope of 20° to 30° below the horizontal.

Average charges for bank holes in medium-hard rock are about as follows:

Average charges for bank holes in medium-hard rock.

Depth of hole.	Sticks of dynamite in primer.	Charge.		
		40 per cent strength dynamite.	Trojan powder No. 2 (granulated).	Stemming.
Feet.		*Pounds.*	*Pounds.*	*Feet.*
28	1 and 2	138	100	24
40	1 and 2	....	267	36
50	1 and 2	170	200	40
70	3	....	550	60
[a] 50	2	[b] 375	....	30

[a] Driven into soil.
[b] FF black powder.

Average toe-hole charges for medium-hard rock are:

Average toe-hole charges for medium-hard rock.

Depth of hole.	Number of sticks of dynamite in primer.	Charge.	
		40 per cent strength dynamite.	Stemming.
Feet.		*Pounds.*	*Feet.*
8	1	12.5	4 to 6
18	1 and 2	60.0	12 to 14

The following are average mixed charges from a number of observations:

Average mixed charges.

BANK HOLES.

Depth of holes.	Quantity of mixed charge.	Charge per foot of hole.
Feet.	*Pounds.*	*Pounds.*
28	188	6.72
40	267	6.68
50	369	7.38
[a] 50	[b] 375	7.50
70	550	7.86

TOE HOLES.

8	12.5	[c] 1.56
18	60.0	3.33

[a] In soil and soft rock.
[b] Black powder.
[c] Holes not chambered.

When the holes are sufficiently chambered or sprung and are ready for loading, an electric-detonator primer, prepared as shown in figure 8, C, is carefully lowered to the bottom of the hole. The first part of the charge, varying from 100 to 300 pounds, is dropped in after it. Another primer is then placed in the hole and the remainder of the charge, 75 to 250 pounds, is added (fig. 10). During the process of loading, the charge man frequently drops in his gage, to determine how rapidly the hole is filling and to guard against the clogging of the powder. Often granulated Trojan powder is poured in to fill the interstices between the sticks of dynamite and the fissured places in the rock (fig. 10). Thus air spaces are minimized and the efficiency of the blast greatly increased. When the charge is all placed, screened clayey sand and loam are carefully shoveled into the hole and packed down for stemming.

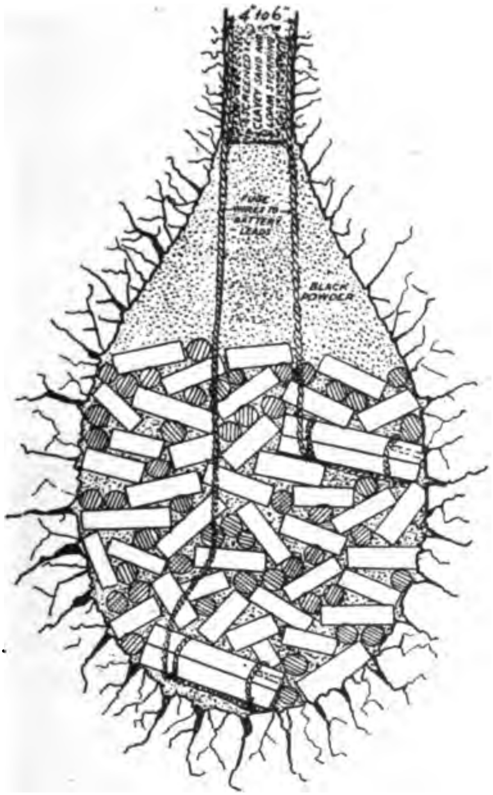
4" to 6"
SCREENED CLAYEY SAND AND LOAM STEMMING
FUSE WIRES TO BATTERY LEADS
BLACK POWDER

FIGURE 10.—Typical blasting charge in bank hole.

All blasting is done at such time as will least interfere with the operation of steam shovels, trains, drilling machines, and general work. Wherever practicable, the blasting is done at the end or before the beginning of the shift or during the noon period. A great deal of blasting is also done by the night squad.

The rules of the company prohibit more than one independent charge in any hole; that is, the explosives must be so placed that no stemming separates any part or parts of the total charge in any hole. Double or triple charges, separated by stemming, are not allowed to be put into one hole, owing to the danger from incomplete detonation. A part of the charge under such conditions might not explode, thus rendering subsequent steam-shovel operations dangerous, for unexploded dynamite is sometimes set off when dug into by steam shovels.

The toe holes are loaded in much the same way as the bank holes, except that the charge has to be pushed into the relatively flat toe holes, a little at a time, with the aid of a charging stick—a long, slender wooden pole (Pl. VII, *A*).

In general the equipment of the charging squad is as follows:

"Springing" gage.
Charging sticks.
Pocket reflecting glass.
Galvanometer.
Rheostat.
Blasting battery.
Explosives.
Fuse.
Electric detonators.
Combination detonator crimper, pliers, and punch.
Wooden mallet.
Large funnel.
Pocket knife.
Wooden axe handle.
Detonators.
Battery lead wire.
Connecting wire.

After the holes have been charged the detonator wires are connected, generally in series, with the wires that lead to the battery (fig. 11). A galvanometer is then attached to the lead wires, and if the whole connection makes a complete circuit the galvanometer needle will be deflected. If there is a break in the circuit it is located by attaching the wires so that first one hole, then two holes, and so on, are left out of the circuit; as soon as a complete circuit is found, it is known that the break must be between the completed circuit and the hole last cut out of circuit. When a complete circuit is established, the wires are ready for connection with the battery. The current used in the testing galvanometer is about one-thirtieth of that necessary to detonate the charge, so that there is an ample margin of safety.

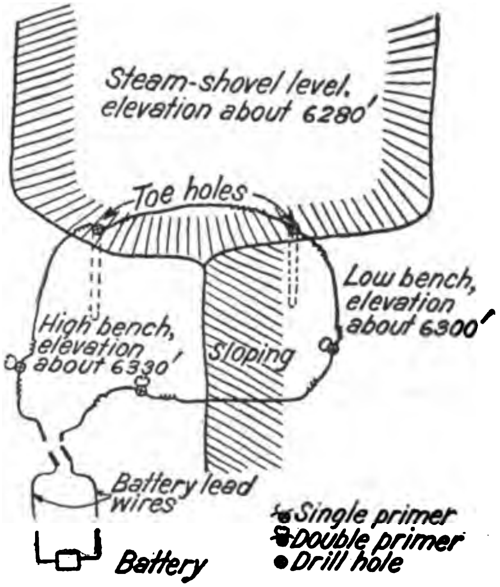
Steam-shovel level, elevation about 6280'
Toe holes
Low bench, elevation about 6300'
High bench, elevation about 6330'
Sloping
Battery lead wires
Battery
Single primer
Double primer
● Drill hole

FIGURE 11.—Arrangement of detonator and electric wires for exploding charge.

Five or ten minutes before the charge man finishes connecting the detonator wires he notifies the engineer of the nearest locomotive or steam shovel, who toots the whistle 40 or 50 times. This warning is understood by all workmen, and they immediately retreat to a safe distance. The final connection with the battery is then made, and after an interval of a few minutes the charge man sets off the round

A. LOADING A TOE HOLE. THE CHARGE IS BEING PUSHED IN WITH A LONG SLENDER WOODEN POLE KNOWN AS A "CHARGING STICK."

B. FACE OF SHOVEL CUT AFTER FIRING BLAST HOLES, SHOWING LARGE BOWLDERS ON WHICH "DOBE" CHARGES ARE BEING LAID.

of shots. After the explosion he notifies the steam-shovel or locomotive engineer, who, when he returns to his post, sounds a long blast of the whistle, indicating that the danger is over.

If a charge misses fire, of course no attempt to withdraw it is made. Such a proceeding would be entirely too dangerous. The remedy is to drill a new hole within 6 or 8 feet of the unexploded one. The new hole must not be so close to the old one that deflection may cause it to break into the unexploded charge. The distance is gaged by the charge man who chambered the old hole and knows the size of the chamber, and by the driller who drilled it. During the drilling of the new hole, as a precautionary measure, nobody is allowed to work in the vicinity of the unexploded hole except the men necessary to operate the drill.

FACTORS AFFECTING BLASTING EFFICIENCY.

The quantity of material loosened by a given amount of explosive depends on many factors, chief among which are: Toughness of the rock; degree of jointing and fissuring; number and extent of free faces of the bench to be blasted; depth and spacing of the holes—their distance apart and from the free faces; character of chambering and of charging of the holes; character of detonation of the charges—whether they are all detonated simultaneously.

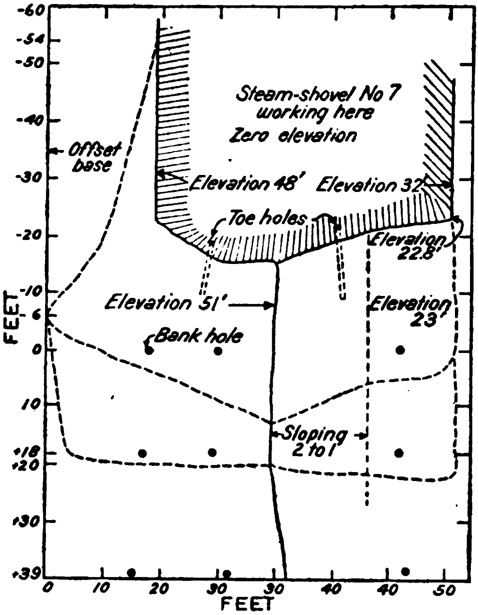

FIGURE 12.—Results of firing bank and toe holes in positions shown. Arrangement of holes and superficial area cut by each series are indicated by dotted lines.

A factor of special significance in determining the charges to be used is the degree of solidity or "tightness" of the rocks at the base or toe of the blasting face. Where the rocks are solid at the toe, toe holes with fairly heavy charges greatly increase the efficiency of the blast, and cause the rock to break down to an even surface. Data regarding several bank and toe holes projected are shown in figure 12. The conditions seemed to be about average for the mine. The results from the blast with these holes were as follows: About 3.2 cubic yards of soil and weathered surface rock was broken per pound

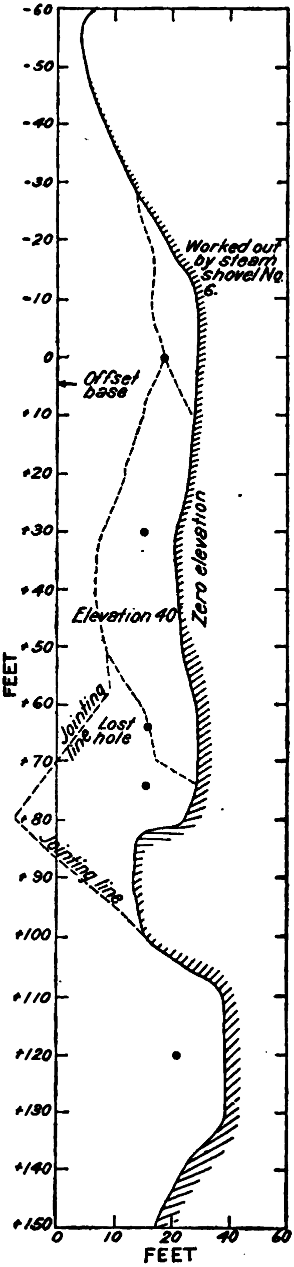

Figure 13.—Results of firing blast holes in firm rock. Shows arrangement for single-line bench holes. The areas cut by each are indicated by dotted lines.

of black blasting powder. Approximately 3.1 cubic yards of somewhat weathered dioritic rock was averaged per pound of Trojan powder. Where this rock was firmer and more solid the results averaged about 1.5 cubic yards for a pound of Trojan and 40 per cent strength Repauno gelatin dynamite (fig. 13). In the tougher areas only about 0.4 cubic yard was broken per pound of Trojan powder. Average results from a number of blasts are given in the following table:

Average results from several blasts.

Character of rock and ore.	Depth of holes.	Explosives used.	Rock broken.	Rock broken per pound of explosives.
	Feet.	*Pounds.*	*Cubic yards.*	*Cubic yards.*
Soil and weathered rock.............	50	378	1,222	3.233
Partly weathered dioritic rock....	70	700	2,168	3.097
Do.............	28 and 50	2,235	3,249	1.454
Relatively soft porphyry.......	40	825	2,264	2.744

For a three-month period in 1913 the average quantity of ore and rock broken per pound of explosives used was 2.55 cubic yards. For a corresponding three-month period in 1914 the average was 2.67 cubic yards. The quantity of both kinds of explosives used during these periods was as follows:

Quantity of explosives used in blast holes during two three-month periods.

Kind of explosive.	Three-month period in 1913.		Three-month period in 1914.	
	Quantity of explosive used.	Per cent of total.	Quantity of explosive used.	Per cent of total.
	Pounds.		*Pounds.*	
Black powder	192,870	44.56	136,950	39.71
Higher explosives.......	240,000	55.44	207,950	60.29

SECONDARY OR "DOBE" BLASTING.

When the bank holes are exploded, 5 to 15 per cent of the material broken is so coarse (Pl. VII, *B*) that it has to be blasted again in order to be handled easily by the buckets of the steam shovels, which have a capacity of 3 to 3½ cubic yards. This secondary blasting is done by placing a number of sticks of dynamite and a primer, with 42 inches of fuse attached, on the rock to be broken, the size of the charge being regulated by the judgment of the powder man. Formerly it was the general practice everywhere to cover charges of this kind with a stemming of clay or adobe, giving rise to the term "adobe" or "dobe" blasting. This manner of breaking rocks is also referred to as "bulldozing."

When the "dobe" charges are all set, the nearest steam whistle toots a warning for the workmen to get out of the way. The chargeman and one or two assistants, each using a piece of fuse equal to the length of the primer fuses (42 inches) as an igniting medium, light the primer fuses and then run to safety. A series of cuts half through the igniter fuse indicate to the men doing the igniting just how fast their fuses are burning and how much time they will have to finish the lighting and get to safety. The number of "dobe" shots placed is counted, and the number of explosions heard is carefully observed so that any missed or delayed shots will be detected.

Prior to the installation of the plant for crushing coarse material, the cost of blasting bowlders, or of "dobe" shooting, was about 1.54 cents per ton of ore loaded. It is estimated that the new crushing plant saves 80 to 90 per cent of that cost.

BLASTING COSTS.

During a three-month period in 1913, when the mine was working at normal capacity, the cost of explosives was 3.75 cents per cubic yard of material loaded. For a corresponding period in 1914, when operations were conducted at only about 60 per cent capacity, the charges for explosives had dropped to 3.56 cents per cubic yard. The difference was due largely to the fact that fewer bowlders had to be blasted after the coarse crushing plant had been installed.

The average cost per pound of all the explosives used in 1913 was 9.55 cents, and in 1914 it amounted to 9.64 cents. "Dobe" blasting is an inefficient method of breaking rock, because the charge is not confined and much of its energy is lost; also because the loading operations of the nearest shovel or shovels must cease while the "dobe" charges are being placed and fired.

BLASTING ACCIDENTS.

Under normal mining conditions about 1,500,000 pounds of explosives is used annually by the Chino company. These figures are so large that, in comparison, the accidents have been few. During 1914 only two men were killed as a result of blasting operations. They miscounted the number of "dobe" shots lighted, and walked back before the last one, which was delayed, had exploded. In 1913 three men were killed. A party of five men had taken refuge under the back end of a steam shovel, against the general orders of the company. The blast was so heavy that the concussion moved the shovel back on the men, fatally crushing three of them.

SAFETY RULES.

To help safeguard its employees the company has posted rules, printed in English and Spanish, so that everyone, especially those that handle explosives, may become familiar with them. Some of the principal rules so posted follow:

Before exploding a blast, care should be taken to give the blasting signal and to wait until every person has reached a point of safety. Do not explode a blast until at least three minutes have elapsed from the last note of warning.

The powder "whistle off" signal must be given after each and every time of blasting.

All persons are warned not to return to the vicinity of "dobes" until at least one-half minute has elapsed from the last shot heard.

When dobying (bulldozing) is done directly in front of a shovel no one should remain on or under that shovel for protection.

In firing bank shots or toe holes the battery wires must not be connected to the battery until the person immediately in charge walks back to the battery and gives the word to connect.

Do not smoke while handling explosives, and do not handle explosives near an open light.

Do not carry loose detonators or electric detonators in the clothing. Carry them in special boxes.

Do not transport caps or cartridges containing caps to the blasting place with the supply of dynamite to be used, and do not place them side by side until ready for explosion.

Do not tamper with caps. If the caps are damaged, discard by loading into some bank hole.

Inspect the place where bulldozing has been done before work is resumed. Recover all powder and caps possible and return to their proper places.

Carelessness will not be tolerated. The careless laborer imperils not only himself but he endangers his fellow workman as well.

All blasting machines must be tested with rheostat at least twice a week. If blasting machine is defective, this must be reported. The machine must not be used until it has been repaired.

During cold weather no dynamite but the nonfreezing powder shall be used unless by order of the general foreman.

Powder boxes and powder cans must not be opened otherwise than with the wooden tools provided (which are mallet and axe handle).

Black powder cans should not be rolled down the bank.

Black powder should not be opened near a light or fire.

Dynamite must not be rolled down the banks tied to a rope except in places and manner designated by the head powder foreman.

It is absolutely prohibited to store powder or caps anywhere about a steam shovel. All such recovered material must be gathered up and returned to the boxes.

Do not allow explosives to become scattered. Immediately after each finished period of loading all unused powder and caps must be returned to their respective places.

Detonators and powder must not be stored in the same place.

Fuse, firing machines, and all tools necessary for blasting must be kept in a box separate from the powder. All material must be kept under lock and key.

Place all powder boxes so that the cartridges will rest in a level position.

Do not fasten a detonator with the teeth or by flattening with a knife; use a crimper.

Do not explode a charge to chamber a hole and then immediately reload the hole; the hole will be hot. Water must be used to cool it.

Do not put two independent charges of explosives in the same drill hole.

Do not attempt to draw nor to dig out the charge in case of a misfire.

No person shall blast a hole alone; he must have some one near him.

For dobying, fuse must not be less than 30 inches; for springing holes not less than 18 inches.

All electric detonators must be tested with galvanometer before placing stemming in the hole.

Not more than three men are to load a hole at one time.

In loading bank holes one primer must be placed in the hole before adding any of the charge of powder.

After primer or powder has been placed in the hole no metal shall be used either to tamp or clear the hole.

All tamping used in holes shall be screened dirt.

Caps and electric detonators shall be stored in separate box, which shall be marked "Cap Box." Nothing else shall be stored in these boxes.

One man is never to trim a bank alone; at least two men should work together.

While trimming, bank men are not to go down the bank unless with a rope securely fastened above.

"GOPHER" BLASTING.

At the present stage of development "gopher" blasting has been practically discontinued. However, in the past it was advantageously used to break down some high banks that were difficult to drill and load in the ordinary way. A good account of one of the largest gopher blasts has been written by R. I. Kirchman,[a] bench foreman for the Chino company. It is so instructive that extensive quotations from it follow. Kirchman first explains that in the preliminary work at Santa Rita, owing to the uneven surface of the ground, the height of the benches varied from a few feet to 150 feet or more. Some of the high benches were maintained because the expense of opening intervening levels was not warranted by the conditions. Locally the rock was considerably fissured and jointed with a tendency to "hang up." Hanging brows of the rock, where the bank was 75 to 100 feet high, came down at uncertain intervals, sometimes endangering the steam shovels near the base. When toe holes were sprung they often caved, thus rendering them useless and necessitating new holes. Because of these difficulties it was decided to handle the bank with a large gopher blast. Kirchman describes the operations as follows:

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[a] Kirchman, R. I., Gopher blasting: Colorado School of Mines Mag., vol. 4, May, 1914, pp. 103-107.

> Three adits were determined upon as shown in figure 14. The north adit was numbered 1, the middle 2, and the south 3. Adits 1 and 2 required two tunnel sets each. These sets were lagged. Adit 3 was not fractured as much as 1 and 2 and did not require timbering. The cross-section dimensions were 3 feet by 5 feet high, driven part by hand drill and part by air-drill machine.
>
> The muck from the main adits was dumped directly in front and around their entrances. The broken earth from the crosscuts was thrown back and leveled to a height just sufficient to allow the passage of a man—that is, an opening about 2 by 3 feet. The excess muck was carried to the entrance. The corresponding crosscuts were thus left half full of muck back to the main adits.
>
> The ends of the crosscuts were chambered to a size sufficient to accommodate the powder predetermined by the amount of yardage over the "gophers." The gopher was assumed to break from a line 10 feet beyond the breast of the main adits, and along a 1-to-1 slope to the surface.
>
> The Mexican laborers were divided into classes of miners and muckers. The miners were paid at the rate of $2.75 per day of 9 hours and the muckers $2 per day. At the start only two 9-hour shifts were employed, but as greater progress was desired three 8-hour shifts were put on.
>
> The latter change left 2 men in each face, or a total of 18 men per 24 hours. The progress was about 4 feet per shift. The total linear feet of gopher driven was 352. The time occupied in driving was 31 days. Loading commenced immediately after completion and required 3 days of 10-hour day shifts, and was accomplished by 4 powdermen and 10 laborers for loading and tamping.
>
> On the bottom of the drifts a walk was laid, composed of single boards placed loosely end to end; no nails were used. The powder boxes were slid along this path to the chambers. Ordinary pocket electric flash lights gave fair light for about 24 hours.
>
> Crosscut 1 was loaded first. The ends of the powder boxes were opened and placed end to end. The boxes were pyramided in a single tier down the middle of the pocket. An electric fuse was placed in the middle box of each layer of the tier, making three fuses to the chamber. Extreme care was taken not to injure the light fuse wires. An important precaution to observe in loading these initial charges is the avoidance of undue pressure upon the fuses or stick powder. The stick powder used was Du Pont 40 per cent Repauno gelatin. The fuses were Victor No. 7, 10-foot lengths, having a resistance of 1.2 ohms.
>
> The black powder, in 25-pound cans, was next placed about the powder boxes. About three-quarters of the black-powder charge was loaded from the breast to the front of the charge and one-half way up to the back. Judson powder, in 12½-pound paper bags, was placed upon the powder cans as compactly as possible. This latter charge filled the pocket clear to the back. The final face of the chamber was sealed with the remaining one-quarter of black powder in cans. The latter precaution was taken to avoid any danger or damage to the dynamite and Judson powder when tamping.
>
> All connecting fuse wires were firmly contacted. The charges were connected in series with No. 14 lead wire, and the fuse wires were wrapped around the lead wires. All fuses were tested with a Du Pont galvanometer, which shows the presence of a circuit and resistance contained therein. All completed circuits in the different chambered pockets were tested before the chambers were sealed with carefully placed

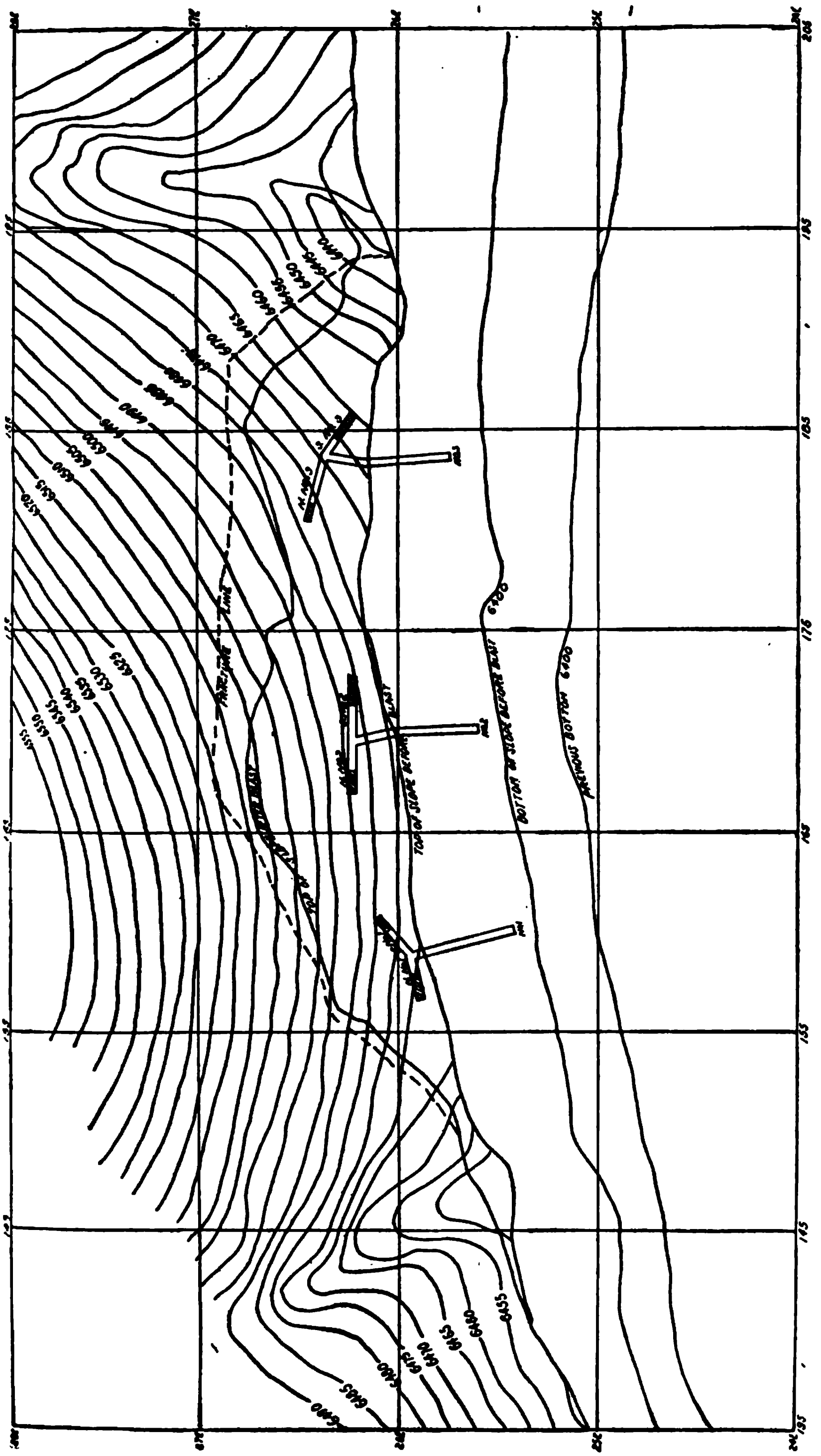


FIGURE 14.—Placing of "gopher" holes for large blast.

muck. The lead wires from each chamber were laid on top of the muck and in one corner of transverse cross section. Pieces of wood broken from the refuse powder boxes were placed at an angle above these wires, thus affording a protection from the rocks. These lead wires were uncoiled as fast as the tamping proceeded.

The tamping was a slow and laborious process. The muck had to be carried in powder boxes and slid along the board path, and dumped into the receding filled faces. At intervals of 3 feet of fill the circuits were tested in all drifts.

During the loading of the gopher the men suffered from powder headaches. These were sharp and annoying, but soon passed away in most cases when brought to the fresh air. The tamping progressed more rapidly when the main adits were reached. An unusual source of danger occurred for a few days during the presence of electrical storms. The precaution of keeping all lead wires well within the adit entrances was observed.

The lead wires from the several entrances were then connected in series. A pair of main lead wires was then laid a distance of 1,500 feet north of the entrance of adit 1. These were not connected until the proper time for blasting. The blasting battery, a Du Pont 1–75 fuse machine, was cleaned and tested.

The final circuit was tested with the galvanometer and a resistance of 24 ohms noted; this indicated a good connection. The eighteen 10-foot fuses used should have had a total of 21.2 ohms; the remaining 2.8 ohms was apparently due to imperfect connections. The resistance of the lead wires was practically zero. On June 25, 1913, the gopher was fired. Some bowlders were thrown 400 to 600 feet by the concussion of the sharp blast. The main mass of the material was not thrown over 200 feet.

The yardage of the gopher was figured on a 1-to-1 slope. The steam shovels removed 60,000 cubic yards (in solid), but the total has not yet been removed. It is estimated that 40,000 cubic yards, in solid measurement, of loosened material is still to be removed.

It was the opinion of some that the gopher was loaded a trifle too heavy. For the class of overburden a load of 1 pound of powder for 3 or 3½ cubic yards (solid) would have yielded better results; but as an experiment for future reference this was a successful gopher. No serious delay occurred, no damage was done, and the practicability of a gopher bank was demonstrated.

The data were summarized by Mr. Kirchman as follows:

Data regarding one of largest "gopher" blasts used by Chino company to break down high bank.

Chamber.	Powder loaded (pounds).					Rate loaded.		
	Black powder.	Judson.	Forty per cent gelatin.	Ratio of explosion to ignition charge.[a]	Total powder charge.	Engineers' estimate.	Pounds per cubic yard.	Cubic yards broken per pound of explosives.
						Cu. yds.		
N. No. 1	7,725	2,400	800	12.65	10,925	25,664	0.41	2.34
S. No. 1		5,500	500	11.00	6,000	14,948	.40	2.49
N. No. 2		5,250	600	8.75	5,850	18,122	.32	3.10
S. No. 2		9,250	800	11.56	10,050	25,197	.40	2.50
N. No. 3		7,050	500	14.10	7,550	12,446	.67	1.65
S. No. 3		9,050	900	10.05	9,950	22,283	.45	2.24
Total or average	7,725	38,500	4,100	11.27	50,325	118,660	.425	2.36

[a] These data are of interest in that they indicate the proper ratio of the ignition charge to the explosion charge. An idea of the ratio of the overburden in cubic yards to the powder in pounds is suggested. A large ratio in this character of ground is recommended.

LOADING ORE.

After the ore and waste have been broken by blasting they are loaded onto cars by Marion steam shovels. Of these, there are six of the 91-ton model, two of the 92-ton model, one of the 100-ton model, and one of the 40-ton model—ten in all.

The 40-ton and the 100-ton shovels are used for digging first or pioneer cuts, also called "thorough" cuts. The 40-ton machine has a large arc of swing to its boom, so that it can carry a cut into a bank, swinging its dipper back to one side far enough to dump into a car close to the side of the shovel and back from the working face of the cut. The other shovels when making first, or pioneer cuts, have to load onto cars that stand on the bank above, outside the excavation that is being made. The 91-ton and the 92-ton shovels are designed to have a maximum lift of 19 and 21½ feet, respectively; hence, in general, they can not make a first cut more than 10 to 12 feet below the level of the loading track. The 100-ton shovel, however, is built with a long boom and dipper arm, so that it has a maximum lift of 35 feet. Its function is primarily to make pioneer or "thorough" cuts, which the other shovels enlarge. It can cut a wide ditch about 22 feet deep, lifting the material up onto cars that stand on the banks outside the cut. It has a dipper with a capacity of 3 cubic yards, whereas the capacity of the 91-ton and of the 92-ton shovels is 3½ cubic yards. This smaller dipper is necessary because the longer boom and dipper arm—92-foot extreme radius—greatly increases the strain on the machinery.

The crew of each shovel consists of an engineer at $190 per month, a craneman at $135 per month, a fireman at $3.50 per 10-hour shift, an oiler at $2.25 per shift, and 6 to 8 pitmen at $2.25 per 10-hour shift, who assist in loading operations and in moving the shovel.

The material handled per day of two 10-hour shifts by each shovel, except the small 40-ton machine, is 1,400 to 2,800 cubic yards, measured in the solid. This rather wide variation is due to occasional shortage of cars, local difficulties of digging, or temporary breakdowns.

The coal, oil, and cotton waste used per 10-hour shift is as follows:

Coal, oil, and cotton waste used per 10-hour shift.

Material.	Price.	Amount used by different sized shovels.		
		40-ton.	91-ton and 92-ton.	100-ton.
Black oil	$0.14 per gallon	3 pints	2 quarts	2 quarts.
Cylinder oil	.37 per gallon	3 pints	3 quarts	1½ gallons.
Engine oil	.22 per gallon	½ pint	1 pint	1 pint.
Crude oil	.10 per gallon	1 quart	2 quarts	1½ gallons.
Cup grease	.05 per pound	½ pound	½ pound	½ pound.
White cotton waste	9.39 per hundredweight.	1 pound	1 pound	1 pound.
Run-of-mine coal	4.30 per ton	3½ tons	5½ tons	7½ tons.

Heavy castings and repair parts for all the shovels are kept on hand so that a shovel can, without much delay, be almost rebuilt in the company's machine shops.

The cost of loading is in general 6 to 12 cents per ton. In a few cases, owing to exceptionally unfavorable circumstances, the loading costs for one shovel for a month have averaged as high as 29 cents per ton. On the other hand, exceptionally favorable conditions have at times reduced the monthly average to a little more than 4 cents per ton.

TRANSPORTATION OF ORE.

In the matter of transportation, it was planned to have the loaded cars moved from and the empties moved to the shovels with as little delay as possible. To handle the trains of ore and waste fourteen 42½-ton and seven 50-ton, 4-wheeled Porter locomotives are used. These have cylinders 15 by 24 and 16 by 24 inches. They are each manned by a locomotive engineer, at $4.25 per 10-hour shift and a fireman, at $3 per 10-hour shift. The quantities of oil and fuel used by each locomotive per shift of 10 hours are about as follows:

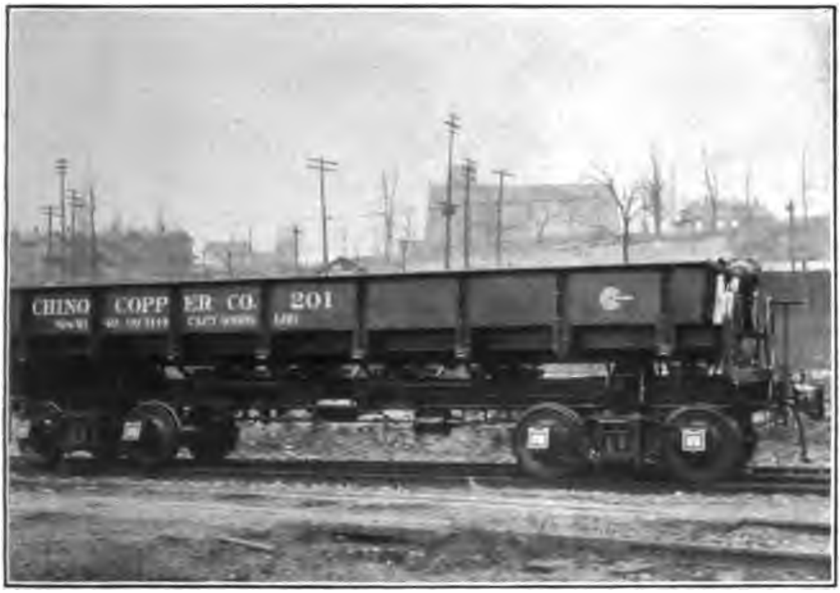
Quantities of oil and fuel used by 42½-ton and 50-ton Porter locomotives with side or saddle water tanks.

Engine oil	1½ pints, at 22 cents per gallon.
Cylinder oil	1 pint, at 37 cents per gallon.
Cup grease	½ pound, at 5 cents per pound.
White cotton waste	½ pound, at $9.39 per hundredweight.
Run-of-mine coal	3 tons, at $4.30 per ton.

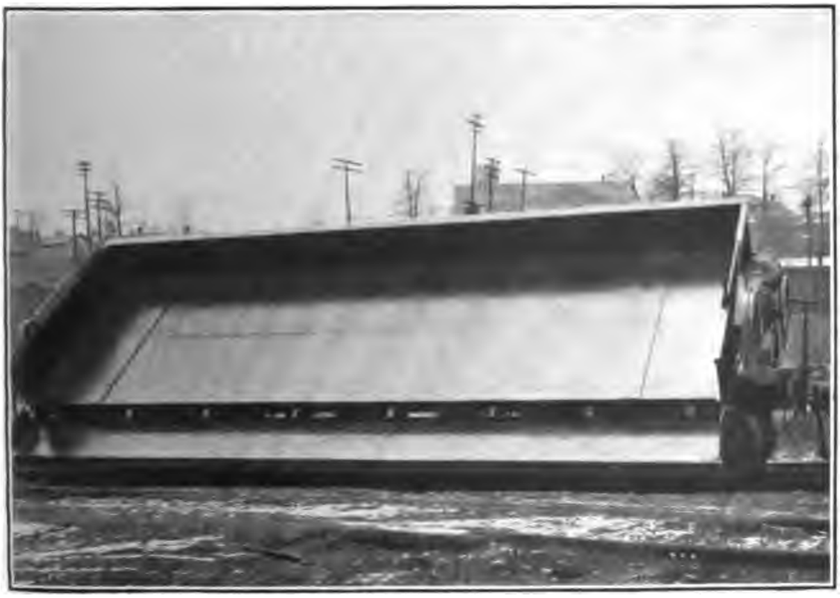
In general, there are always about two locomotives in the shop undergoing repairs. In addition, one locomotive is held just outside the shop with steam up ready for instant service. The total repair, upkeep, and all other expenses of the locomotives amount to about 0.2 cent per ton of material handled.

The ore cars in use are about as follows: Forty-five 12-yard, side-dump, Oliver steel cars; forty 6-yard steel cars not equipped with air brakes; four 20-yard steel cars fitted with air brakes, air dumping appliances, etc. The four last-mentioned cars are leased from the Santa Fe Railroad, and have given such good satisfaction that the Chino company has ordered several of the new Clark 20-yard extension dumping steel cars (Pl. VIII). These dump on either side, by air or by hand power, and are said to be satisfactory.

The experience of the Chino company has been that 6-yard cars are too light for loading with heavy shovels and too expensive to keep in repair. Not being fitted with air brakes, they are rather dangerous to handle on heavy grades. The large, heavy, air-brake



A. STEEL ORE CAR USED AT SANTA RITA. DUMPS ON EITHER SIDE BY AUTOMATIC AIR CONTROL OR BY HAND, CAPACITY 20 CUBIC YARDS.



B. STEEL ORE CAR IN DUMPING POSITION.

and air-dump cars, though expensive, are cheaper in the long run, under the conditions of heavy service that prevail at Santa Rita.

Ore that is fine enough not to need crushing is generally loaded directly into 50-ton steel railroad cars, the property of the Santa Fe Railroad Co., and is sent direct to the mill at Hurley without having to pass through the crushing plant just below the mine.

The following table shows the number of cars of each size loaded per month, during five typical months, and the cost of keeping them in repair. The repair cost per dry ton of ore handled and per cubic yard of waste is also given. The 50-ton cars are the property of the Santa Fe Railroad Co., but are kept in repair by the Chino company while used by them, and so are included in the repair account.

Cost of car repairs for five typical months.

CARS USED IN MINING ORE.

Month.	Cost of repairs.			Number of cars loaded.			
	Labor.	Supplies.	Cost per dry ton.	20-yard.	12-yard.	6-yard.	50-ton.
1914.							
January	$188.81	$196.19	$0.00196	....	100	....	4,028
February	148.99	85.01	.00124	....	4	....	4,066
March	138.48	103.40	.00109	....	898	....	4,435
August	94.93	78.72	.00142	107	2,103	....	1,837
September	86.24	91.87	.00171	12	2,015	....	1,438

CARS USED IN STRIPPING WASTE.

			Cost per cubic yard.				
January	$1,699.33	$1,765.73	$0.01122	....	27,124	15,711	....
February	1,340.88	765.08	.00741	....	29,573	13,043	....
March	1,246.36	930.61	.00629	....	34,525	16,105	....
August	854.35	708.48	.00838	1,017	10,169	10,778	12
September	776.17	826.86	.01008	1,548	9,511	9,065	14

The material loaded onto cars by the steam shovels is hauled out of the pits to one of the following destinations: The mill, direct; the crusher; the ore stock pile; or directly to the smelter. The waste goes to the waste dumps. The lowest grade of ore, containing about 0.8 per cent copper, which has to be moved to get at other higher grade ore and to develop the workings properly, is sent to the ore stock pile if the copper market happens to be unfavorable. It is left there until such time as it can be most favorably utilized. Only the ore that contains many bowlders too large to pass the 12-inch by 13-inch grizzly at the mill is sent to the crusher. The other ore is sent direct to the mill, except the occasional carloads that average high in copper; these are shipped direct to the smelter.

The grade of the pit tracks varies considerably. The average grade of all the tracks to the waste dump, the stock pile, the crusher, and the railroad yards is estimated to be 2¼ per cent. Some idea of the amount of material handled over the company's tracks during the month of July, 1914, when the mine was working at normal capacity and during the month of September, 1914, when it had greatly curtailed its output owing to the European war, is given in the table following:

Data showing material handled on Chino company's tracks.

Month.	Ton-miles of haul.					Grade miles, haul to waste and ore piles and to crusher.
	Waste.	Ore piles.	Crusher.	Mill or smelter.	Total.	
1914.						
July	953,055	39,911	....	228,771	1,221,737	2,533
September	447,486	3,045	36,517	78,830	565,878	2,055

The figures in the second, third, fourth, and fifth columns of the above table are obtained by multiplying the average length of haul by the tonnage moved, thus giving the ton-miles; the figures in the last column are the product of the linear miles and the rise in feet per mile for hauls to the waste and ore piles and to the crusher. The cost of hauling out and dumping waste averaged a little less than 3 cents per cubic yard.

The tracks are of standard gage and consist of 60-pound rails laid on 7-inch by 9-inch ties 8 feet 2 inches long and spaced 20 ties for each length of 33-foot rails. A few 75-pound rails are used in the pits. The Chino company constructed at its own expense and is operating about 24 miles of railroad track. This trackage connects with the railroad yards of the Santa Fe branch line to Santa Rita. It is provided with ample turnouts and crossovers and is systematically operated under the supervision of a yardmaster and his assistants, switch tenders, etc.

The maintenance of the pit tracks is under the supervision of an assistant roadmaster. Subordinate to him are 4 track foremen, each in charge of a gang of 13 men. These gangs shift track and keep the track lines up to the steam shovels. The waste and ore-dump tracks are looked after by 7 track foremen, each in charge of a gang of 13 men. These crews level off and trim down the dumps and keep the tracks shifted out toward the edge of the dump slope. The pay of the laborers is about $2.05 per 10-hour shift. The track foremen receive $4.55 per 10-hour shift.

CRUSHING OF COARSE ORE.

The coarse ore grizzlies at the Hurley mill have openings 12 by 13 inches. It is intended that the ore shipped to the mill shall have few fragments too coarse to pass through these openings. Up to 1914 ore fragments coarser than this had to be broken by "dobe" blasting, or by hand, either at the shovel, on the cars, or at the mill. These methods of breaking the large fragments were relatively expensive. It was therefore decided to build near the mine a plant for crushing coarse ore, the plant to accommodate any bowlder that could be handled by the 3 to 3½ cubic yard dippers of the steam shovels.

A site for the new crushing plant was selected on a side hill, so that the ore cars could be run on a track about 50 feet higher than the feeding hopper of the large crusher (Pl. IX, *A*). It is situated about 1¼ miles below the mine, and is convenient to the branch line of the Santa Fe Railroad which leads to the mill at Hurley. The cars of coarse ore from the mine are dumped onto the steel grizzlies (Pl. IX, *B*), the bars of which are about 8 inches apart. The oversize pieces roll down into a hopper that feeds to a 48-inch by 60-inch Superior jaw crusher. This vast crusher weighs 100 tons, and is driven by a 150-horsepower motor. It crushes to 6 inches and smaller, and has a capacity of 3,000 to 5,000 tons per 10 hours. A 48-inch overlapping steel pan conveyor, driven by a 50-horsepower Westinghouse induction motor, conveys the crushed product, and the fines that have gone through the grizzly bars (Pl. IX, *B*), up a 15½° incline to the top of the loading bin (Pl. IX, *A*). A number of observations showed that one-fortieth to one-fourth of the contents of each car dumped onto the grizzly went over into the crusher. The remainder went through the bars as undersizes or fines.

The loading bin, of steel, is 40 by 14½ feet at the base, and 23 feet high. The upper 9 feet of the longer dimension tapers in to 26 feet 8 inches instead of 40 feet as at the base. It has a capacity of 567 tons, on the basis of 21 cubic feet to the ton of ore. The bin has six loading chutes on each side, and is between two tracks, so that two large steel cars can be loaded at the same time. The loading chutes are high enough above the rails so that the loading is all by gravity. From 10 to 20 minutes is required to load a car having a capacity of 100,000 pounds. The coarser the material the easier the loading, after it is once started. Fine material sometimes chokes the chutes, until prodded loose by the bars of the car loaders.

About four 12-yard cars at a time are pushed by a dinkey locomotive from the mine to the up-grade side of the grizzly. The locomotive engineer then gives three blasts of his whistle. If the crusher man is ready for more ore he answers with two whistles. The first car is set opposite the grizzly and dumped by hand, then the other cars are set and dumped as fast as the crusher can take the ore. After all the cars in a trip have been dumped, the locomotive engineer sounds two blasts of his whistle, and pulls the dumped cars past the grizzly; after the cars have been uprighted and fastened, he proceeds to the mine for more material.

Five men are required at the top of the grizzly to dump and upright the cars. A trip of 4 cars can be dumped and uprighted in about 5 minutes. After the train has pulled out, the men shovel onto the grizzly any material that has fallen aside. The crew operating the crusher consists of a foreman, two mechanics, two crusher runners, four car men, one loader, and three laborers. The total operating wages amount to about $35 per 10-hour shift.

The total cost of building and equipping this crushing plant was about $127,000, of which $42,000 was for labor and $85,000 for supplies.

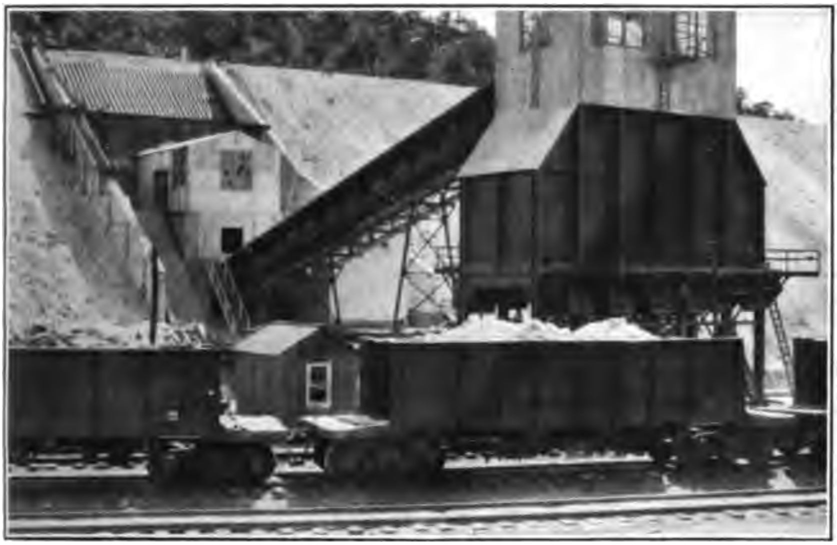
The total cost of operating the plant for crushing coarse material for a part of August, 1914, was $1,231.84, or at the rate of 1¼ cents per dry ton of ore. For September, 1914, the total operating cost of the plant was $1,994.84, or at the rate of 1.9 cents per dry ton of ore. This higher cost was due to some important reinforcements required for the retaining walls and additional equipment charges.

MINE DRAINAGE.

The precipitation in the Santa Rita region is so light that the amount of water that gets into the workings is relatively small. The slopes in some of the open cuts are such that they drain directly to the Santa Rita Creek. In the deeper open cuts sumps with electrically driven pumps take care of the water. The 300-foot level of the underground workings, which are old workings kept open mainly for drainage and prospecting, is drained by two Aldrich triplex pumps and one Byron-Jackson centrifugal pump, all operated by electric power. These pumps have a total capacity of about 1,000 gallons per minute, against a head of 400 feet. A reserve pumping plant on this level consists of two Cameron pumps, 16 by 7 by 18 inches. They are operated by compressed air, and are held in readiness in case of accident to the electric pumps or to the supply of electric power. On the 400-foot level the small amount of seepage is collected in a sump, and pumped to the 300-foot level by a 16-inch, three-stage, electrically driven Byron-Jackson centrifugal pump, having a capacity of 900 gallons per minute.

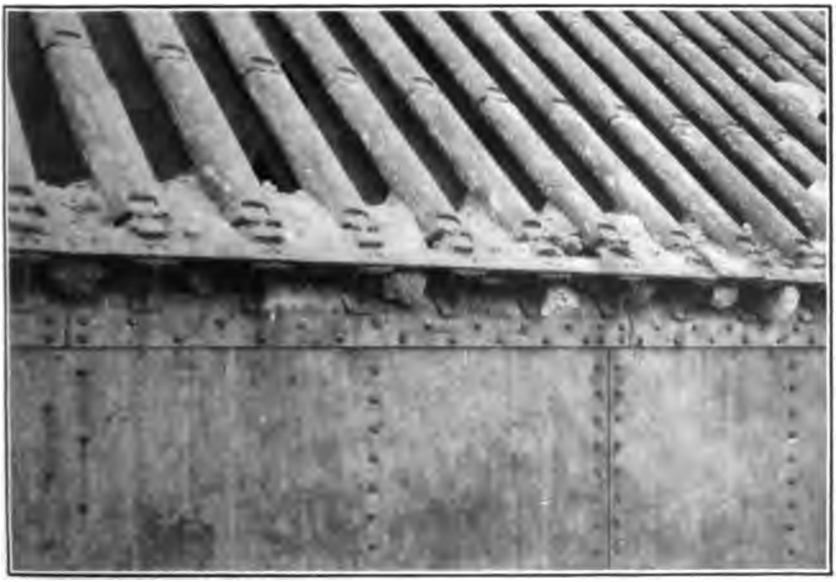
EFFECT OF SLOPES OF OPEN CUT.

The stability of the slopes of an excavation has an important bearing on the cost of the excavation. Where the slopes stand steeply the cost is much less, other things being equal, than where they slide to flat angles, owing to softening by ground water or other causes. If the slopes of the excavations at Santa Rita were as unstable as, say, those of the Culebra Cut, much would be added to the cost of mining.



A. PLANT FOR CRUSHING COARSE ORE, AND LOADING BIN, SHOWING CARS AND LOADING TRACKS.

The coarse material is hauled to this plant over the upper track, the grade of which is visible in the upper part of the picture, and dumped over the grizzly, from which the oversize goes to the crusher situated in the structure below and in front of the grizzly. An inclined belt conveyor with overlapping steel pans elevates the fines from the crusher and grizzly to the loading bin, from which the cars are loaded on the lower tracks.



B. VIEW OF PART OF THE GRIZZLY SHOWN IN PLATE IX, *A*. THE OPENINGS BETWEEN THE STEEL BARS ARE ABOUT 8 INCHES WIDE.

At Santa Rita great open cuts have been made and from these ore and waste are being excavated by steam shovels. These vast open pits are thus constantly being widened, from the central area outward, and are being deepened to the bottom of the ore body. The steam shovels operate on benches, as already explained, one or more to a bench, depending on the location of the ore bodies, the requirements of mining, etc. The bench levels, in this process of being widened, are cut back into the rising ground, and into the sides of the hills, necessitating slopes that, locally where ridges are cut, are several hundred feet high.

These high slopes, in places, may be largely in waste material that overlies valuable ore bodies; hence their stability has much to do with the cost of mining. If they stand steeply, much less excavation is necessary than if they slough down to flat grades. At Santa Rita the open-cut slopes have stood remarkably well, chiefly because of the following factors:

(1) There is little ground water in the soil, which covers the rocks to depths of a few inches to 30 feet or more. This is because of the relatively small precipitation and the good drainage conditions. The latter are due to the porosity of the soil and the slope of the ground toward the open cut.

(2) The upper or weathered zone, which varies much in thickness, depending on the character of the rocks, the degree of jointing, etc., is considerably cut by jointing (Pl. X), and some shearing. This weathered zone, together with most of the mineralized rocks, is locally much altered, not only by weathering, but also by hydrothermal action. The effect on the slopes is as follows:

(*a*) The weathering and mineralizing solutions have much sericitized the feldspars, thus weakening the rocks and causing a considerable degree of pulverization when they are blasted. With excessive ground water this condition would also tend to give them muddiness and mobility. However, ground water being largely absent, as already explained, they are strong enough to stand at fairly steep slopes without crushing or movement, except some sloughing, for all heights of slope that are likely to be encountered.

(*b*) The jointing has cut the rock mass into irregular blocks (Pl. X), and these will, of course, slough off where the slopes are rather steep, especially after they have been somewhat loosened and jarred by blasting. However, this jointing is not great enough to give a flowage-like motion, or "creep," to the jointed material, even on the highest and steepest slopes. Where faulting has sheared the rocks, and slikensided them, there is considerable local sloughing, but such zones are too restricted to cause a noticeable increase of mining cost.

The rocks below the weathered zone are less jointed and are more stable than those just described for the weathered zone; hence they stand fairly steeply.

The steepest slopes of the cut are those in the quartzite and other hard rocks. They range from 55° to 85°, depending on whether the principal joint planes dip toward the excavation. Probably the average slope in such rocks is about 70°, with the soil covering and loose rock on top sloping at about 40°. In the softer rocks slopes of 40° to 70° are maintained. In calculating the yardage of stripping to be removed, a slope of 1 on ½ was first used, meaning one unit vertical to one-half unit horizontal. However, in 1915 the use of a slope of 1 on ¾ was adopted as being safer for making ore estimates.

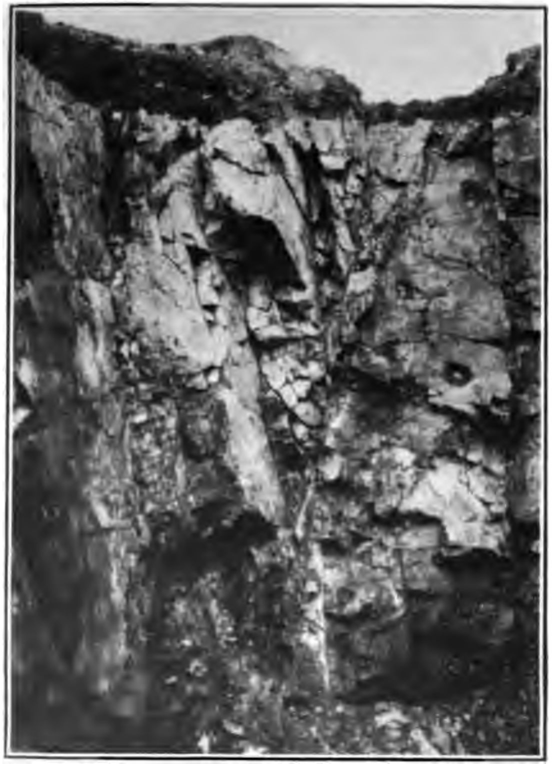
The flattest slopes are those at which the material in the waste dumps comes to rest. Several of the large dump slopes, some showing about 200 feet of elevation were measured, and they all approximated 37°, in spite of the fact that they contained much fine material. The upper half of each of the dump slopes was about 37½° and the lower half about 36½°. The increased flatness below is due to the tendency of the larger fragments to roll down and flatten the slope at the bottom.

COMPRESSOR PLANT.

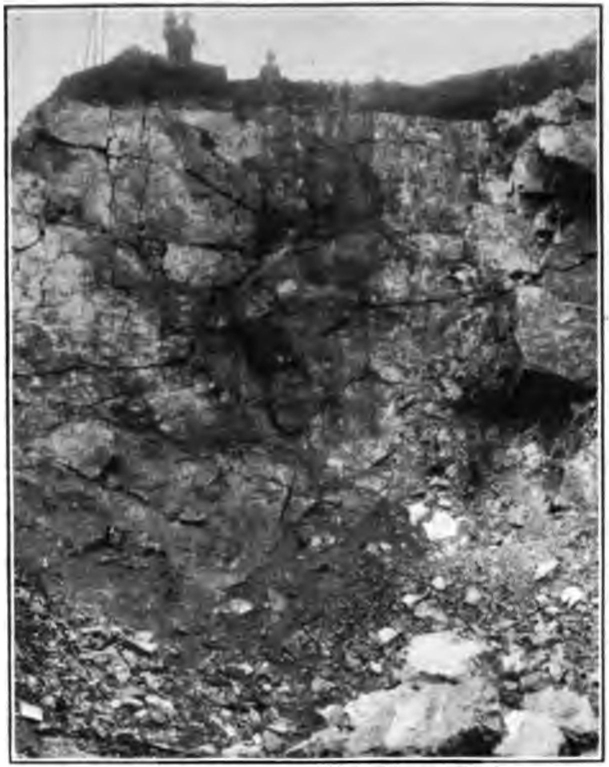
The air-compressing plant contains two Nordberg compound compressors, each driven by a 200-horsepower electric motor. The air, when compressed to 90 pounds per square inch, is stored in two steel cylindrical receivers 4½ feet in diameter and about 13 feet long. One of these is a discarded steam boiler that serves its purpose reasonably well; the other was built as an air storage tank. From these reservoirs pipes distribute the air to the places in the workings where it is needed to operate the tripod and small hammer drills, and the few churn drills that formerly used steam but now use air. The power charges against the compressor are $550 to $650 per month.

CENTRAL POWER PLANT AND POWER DISTRIBUTION.

The Chino company has its power-generating and power-distributing center at Hurley, where the concentrating plant is situated. Power is distributed in the form of electrical energy generated in a large modernly equipped steam-driven plant. When the plant was visited by the authors in 1914, steam was supplied from eight Heine safety boilers, each of 445 horsepower capacity. One or two of these were always in reserve, or being cleaned, or undergoing repairs, while the others were in active operation. Two additional 419-horsepower boilers were being added. The boilers were equipped with superheaters, and with mechanical stokers of the Greene and American types. They used natural draft, except the two new ones, which had forced draft.



A. FACE OF EXCAVATION, SHOWING JOINTING.



B. ANOTHER VIEW SHOWING JOINTING.

The plant operated with steam at a pressure of 180 pounds and heated to 370° F. One fireman and two helpers, per eight-hour shift, operated the plant. The additional boilers called for two more helpers per 24-hour day. In the boiler room were also two boiler washers and one repairman. Feed water heated to a temperature of 190° to 195° F. by Stilwell heaters, and measured by V-notch weirs, is supplied to the boilers by three automatically controlled pumps, each 8 by 12 by 7 by 12 inches.

The boilers supplied steam to three Nordberg compound engines, each 27 by 60 by 48 inches. Each engine drove a 1,250-kw., 480-volt, Allis-Chalmers generator. Steam was also supplied to a 150-horsepower Ballwood tandem compound engine, which drove a 100-kw., 250-volt, Allis-Chalmers generator. Another 100-kw., 250-volt, generator, of the Westinghouse type, was driven by a 150-horsepower Westinghouse steam turbine, and one 100-kw., 250-volt, Allis-Chalmers generator was driven by a 150-horsepower Allis-Chalmers alternating-current motor. There were other smaller motors about the plant. A Worthington surface condenser, operated by steam-driven dry-vacuum and motor-driven wet-vacuum pumps, provided water for each dynamo engine.

The water from these condensers, the condensate, was pumped into a series of baths, in which the cylinder oil and graphite was removed. The cleaned water was then returned to the feed-water storage tanks in the boiler house. The exhaust from the other engines went direct to the feed-water heater. The water for cooling the condensers was circulated by two 16-inch Worthington volute pumps driven by 200-horsepower Allis-Chalmers induction motors operated at 690 revolutions per minute against a pressure of 37 pounds. Only one of these pumps was used at a time, the other being held in reserve.

During July, 1914, the cost for the coal used at the Hurley plant was 33.2 cents per 1,000 pounds of steam generated, and during September it was 32.7 cents.

In the transformer room the plant contained two banks, each of three single-phase, 400-kilovolt-ampere, 60-cycle, Allis-Chalmers transformers, oil-insulated and water-cooled. These transformers could be run in multiple or separately, and they stepped the current from 480 to 24,000 volts.

Attached to the power plant there was an oil-filtering plant for treating used oil. It had a capacity of 25 gallons per minute and a storage capacity of 450 gallons. The used oil drained by gravity to the filtering room and three small double pumps circulated it to the pressure filter tanks. After filtration, the oil was forced by air pressure to the engine room for reuse. All oil pipe lines leading to and from the filter room were colored. For example, yellow indicated engine oil, brown indicated the cylinder-oil drain to the filter, green the crank-pin oil drain, etc. The cylinder oil required two filterings. The cotton waste was cleaned by a centrifugal machine.

During the year 1913 the power-generating plant had a net output of 24,198,633 kilowatt hours. The average cost for the year for current delivered to the feeder circuits on the switchboard was $0.012 per kilowatt-hour, not including the distribution losses. An analysis of this cost is given in the following table:

Analysis of cost of generating power at Santa Rita plant in 1913.

Item.	Total cost.	Cost per kilowatt-hour.
Overhead expense, direct supervision	$6,081.68	$0.000251
Labor, operating	27,970.48	.001156
Labor, repairs	8,864.88	.000366
Supplies, operating	167,649.56	.006928
Repair materials	4,597.14	.000190
Total direct cost	215,163.74	.008890
Overhead expense, general administrative (estimated)	7,600.00	.000314
Taxes (estimated)	5,530.00	.000229
Fire insurance (estimated)	1,383.00	.000057
Interest on plant, $553,000, at 6 per cent	33,180.00	.001371
Depreciation, 4 per cent (amortization fund based on 15-year life, 6.66 per cent depreciation at 6 per cent interest, compounded semiannually)	22,120.00	.000914
Gross cost of generation	284,976.74	.011776

During the month of September, 1914, the Santa Rita operations consumed 105,529 kilowatt-hours. The distribution of power for the month of July, 1914, when the mine was operating at normal capacity, and for September, 1914, when it was operating at about 50 to 60 per cent capacity, was as follows:

Distribution of power in Santa Rita operations during two months.

JULY, 1914.

Item.	Kilowatt-hours.	Electrical horsepower-hours.	Kilowatts.	Electrical horsepower.
Milling	1,521,101	2,039,011	2,044.49	2,740.60
Pumping	478,514	641,439	643.16	862.15
Current for town of Santa Rita	202,246	271,107	271.84	364.39
Shops	12,150	16,286	16.33	21.89
Lights	30,560	40,965	41.07	55.06
Total	2,244,571	3,008,808	3,016.89	4,044.09
SEPTEMBER, 1914.				
Milling	981,331	1,315,459	1,362.97	1,827.04
Pumping	342,971	459,746	476.34	638.54
Current for town of Santa Rita	105,529	141,459	146.57	196.47
Shops	12,150	16,286	16.87	22.61
Lights	22,913	30,714	31.82	42.65
Total	1,464,894	1,963,664	2,034.57	2,727.31

SANTA RITA POWER AND SHOP EQUIPMENT.

A high-tension transmission line from the company's electric power station at Hurley, about 11 miles south, furnishes power to operate the shop machines, compressor, plant for crushing coarse material, pumps, etc., and to light the town of Santa Rita. The line is designed to carry a pressure of 24,000 volts, but this high pressure is stepped down by transformers at Santa Rita to 440 volts for use in motors and to 110 volts for lighting.

In general, the repair plant may be said to consist of a machine shop, with blacksmith and boiler shops attached, and a carpenter shop. At the time of the authors' visit in 1914, the working force of the mine had been considerably reduced, owing to war conditions. At that time the machine shop employed 3 blacksmiths, 4 blacksmith helpers, 1 drill sharpener and helper, 9 machinists, 5 machinist's helpers, 4 boilermakers, 4 boilermaker's helpers, and 9 car repairers. The blacksmiths and machinists were paid $4.50 for 9 hours and the boilermakers $5. The car repairers got $3 to $4 for 9 hours. All the helpers were paid $2.50 to $3 for 9 hours.

The shop equipment was as follows:

Shop equipment of Chino Copper Co. in 1914.

1 Sellers power hammer, operated by air.
1 coke furnace for extra-heavy forging.
2 blacksmith forges.
3 lathes: 1 Davis, 12-inch; 1 Bradford, 18-inch; and 1 Pond, 36-inch.
1 Acme bolt cutter.
1 Williams pipe machine.
1 Barnes drill press.
1 Miles radial drill.
1 Steptoe shaper.
3 emery wheels: 1 wet, 1 dry, and 1 drill grinder.
1 American planer.
1 Long & Allstatler punch and shears.
1 Watson Stillman portable wheel press.
1 6-ton hand crane.
1 power hack saw.
2 Buffalo forge blowers, operated by a Allis-Chalmers 25-horsepower motor.

In the drill-sharpening shop, a No. 3 Leyner drill-sharpening machine, operated by two men, took care of all the steel for the tripod drills. In this shop there was also a boiler flue scaling machine, an oil furnace for heating flues, a flue welder, a flue swedging machine, and a circular saw for cutting wood, all driven by a 15-horsepower motor.

Before the water-softening plant was installed the locomotive and steam-shovel flues had to be taken out and the scale removed about every three months, at great expense. Subsequently the frequency of scaling has been reduced to about once each year. Flues are welded, swedged wherever necessary, and used over again. The composition of the boiler scale taken out was about as follows:

Composition of boiler scale from flues of Chino Copper Co.

Constituent.	Per cent.
Volatile matter	33
Insoluble	13
Al_2O_3	6
Fe_2O_3	14
MgO	1
CaO	28
CO_2	3
S	2
Total	100

Locomotive, steam-shovel, drill, and pump parts were kept in stock. Car axles, car wheels, and all castings were received in the rough and were machined in the company's shops when needed. Locomotives and steam shovels were practically rebuilt on the premises when necessary.

The carpenter shop, a large building, had a lumber yard attached in which about 100,000 feet of material was always kept on hand. A 25-horsepower motor drove a circular saw, a handsaw, a swing cut-off saw, a lathe, and other accessories. Nine carpenters and four helpers were employed in 1914. Carpenters were paid $4.50 per day of 9 hours and their helpers up to $3 per day. Mexican laborers working around the carpenters' shop got $2 per 10-hour shift. The construction and repair of all the company's dwelling houses and other buildings, as well as bridge building, timber framing, etc., came under the charge of the foreman carpenter.

POWER COSTS.

During the month of September, 1914, after the mine work had considerably slowed down, 105,529 kilowatt hours of electrical energy was used at Santa Rita at a total cost of $1,075.31. Of this cost $247.10 was for labor, and $828.21 for supplies. The distribution of the cost was as follows:

Distribution of cost of power generated for town of Santa Rita by Chino Copper Co.

Item.	Per cent.	Cost.
Compressor	40	$430.12
Machine shop	3	32.26
Assaying and sampling	1	10.75
Mine drainage; electric pumps.	6	64.52
Water supply; 3 pumping stations	15	161.30
Carpenters' shop	1	$10.75
Light service, town lights, etc.	8	86.03
Crusher operation	26	279.58
Total	100	1,075.31

This cost amounts to 1.02 cents per kilowatt-hour, or 0.76 cent per horsepower-hour. Assuming that the power was used for 720 hours, the number of hours in the month, the cost per horsepower per month was $5.47.

WATER SUPPLY AT SANTA RITA.

The somewhat arid climatic conditions of the region give particular importance to a water supply adequate to the needs of such extensive mining operations as those at Santa Rita. However, a series of wells and some small streams supply ample water and a water-softening plant eliminates the excess of calcium and other salts and renders the supply satisfactory for domestic and boiler use.

The largest well at Santa Rita is the Pinder. It is near the bed of Santa Rita Creek, and much of its supply comes from the subsurface flow of that creek. A Dean triplex 9-inch by 12-inch electric pump lifts the water from this well and forces it through 3,600 feet of pipe against a difference in elevation of about 410 feet. The diameter of the discharge aperture of the pump and of the first 30 feet of the pipe is 7 inches; the next 2,500 feet is 8-inch pipe, and the last 1,100 feet is 12-inch pipe. The gage on the water pipe near the pump shows a pressure of about 200 pounds per square inch. Against this pressure the pump throws 175 to 200 gallons per minute, more or less. It is operated by an Allis-Chalmers, 75-horse power, three-phase motor, using a 440-volt current. The curvature of the pipe line and the lengths of the different sizes of pipe are shown in figure 15. The well is also equipped with a steam auxiliary plant for use in case of necessity. During October, 1914, 4,965,230 gallons was pumped from the Pinder well to the water-softening plant, or at the average rate of 10,558 gallons per hour of pumping. In addition, 1,449,782 gallons was pumped for domestic purposes from "drill hole I" and 1,440,450 gallons from the Booth well for general supply.

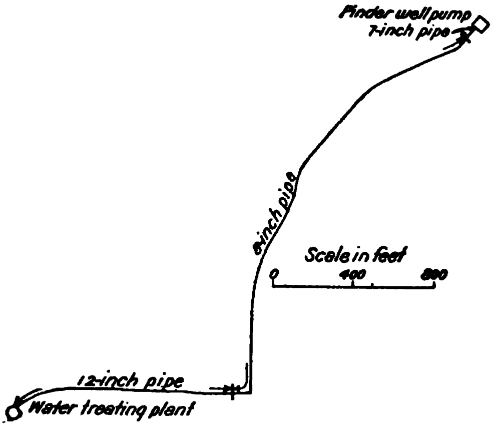


FIGURE 15.—Horizontal curvature and lengths of different sizes of pipe of Pinder well pipe line.

Each blast-hole churn-drill machine uses about 200 to 300 gallons of water per eight hours. The larger prospecting churn drills each use about 500 gallons per eight hours for all purposes. Steam shovels each require 6,000 gallons, and mine locomotives about 4,000 gallons each per 10 hours.

WATER-SOFTENING PLANT.

A Reisert automatic water-softening plant, with a capacity of 9,000 gallons per hour, takes the excess of calcium and allied salts out of the water so as to make it suitable for boiler use. The plant is situated on a hill, about 300 feet above the town and about 350 feet above the mine, where the water is mostly required.

Figure 16 shows the flow sheet of the plant. The raw water is admitted to the bottom of the saturator, where it comes into contact with cream of lime previously supplied from the lime-mixing or slaking compartment. In rising and mixing with the cream of lime, the raw water reaches the top of the tank in a saturated condition and nearly clear. From the lime saturator it flows into the mixing pan at the top of the settling tank, coming into contact there with raw water and with soda ash solution from the soda compartment. The proper proportions of these inflowing solutions are automatically regulated by float valves. After the raw water, the saturated lime water, and the soda ash solution have been thoroughly mixed they pass from the mixing pan through the downtake pipe to the bottom of the settling tank. From there the mixture slowly rises to the top, and the precipitates resulting from the chemical reaction slowly settle out. At the top the water is fairly clear, but it overflows into filter tanks where the last of the suspended matter is removed. Emerging from these, it goes to a storage tank ready for use. The filter tanks are washed out, whenever necessary, by reversing the flow. The solid matter may be drawn from the bottom of the settling tank by opening a valve.

The plant is operated by one man under the supervision of the mine assayer. The mechanical regulation and operation of the plant can be taught to the average operator in a few days.

FUEL.

The fuel used by the Chino company comprises bituminous and subbituminous coals from northern New Mexico. Run-of-mine coal and No. 2 lump are principally used. Under normal working conditions about 63,000 tons is used annually at Santa Rita. The average cost of coal delivered there is $4.35 per ton, more than half of which is for freight charges.

When received the coal is unloaded to storage piles. To keep the temperature down and prevent fires from spontaneous combustion, the piles are built around ventilating stacks, which are wooden conduits a foot square made out of 1-inch or 1½-inch boards in which many holes have been bored (Pl. V, *B*).

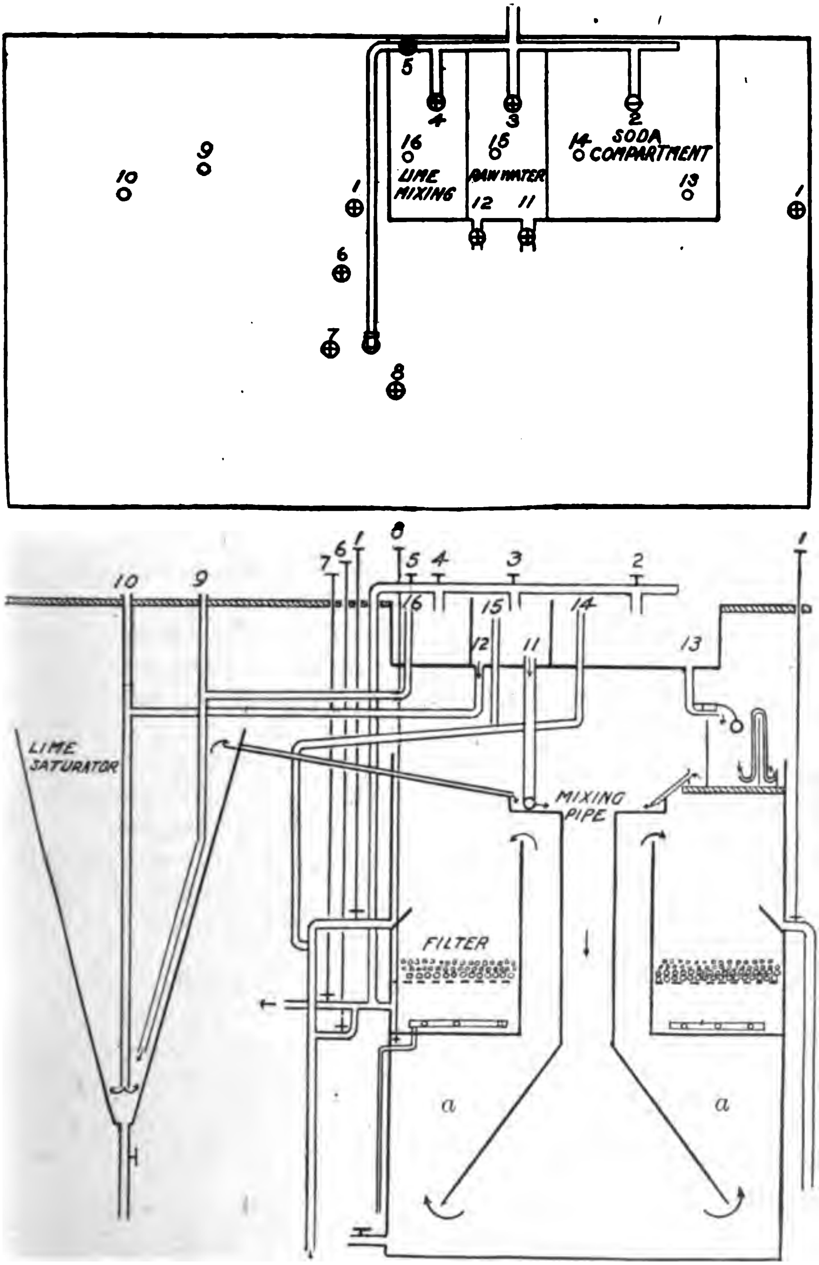


FIGURE 16.—Flow sheet of Santa Rita water-softening plant. *1*, Drains filter to top of gutter; *2*, raw water inlet to soda compartment; *3*, raw-water inlet to raw-water compartment; *4*, raw-water inlet to lime-mixing compartment; *5*, by-pass to reverse flow of water through filter; *6*, drains filter down to tight head; *7*, treated-water outlet; *8*, air valve for cleaning filter; *9*, open pipe for cleaning lime passage; *10*, open pipe for cleaning lime-saturator outlet; *11*, controls flow of raw water through treating plant; *12*, controls flow of saturated lime water; *13*, soda-solution outlet; *14*, drain for cleaning out soda compartment; *15*, drain for cleaning out raw-water compartment; *16*, passage from lime-mixing compartment to saturator; *aa*, *settling tank*.

MINING COSTS.

Economy of production is the policy that, other things being equal, offers the greatest opportunity for success in mining, as in most other lines of business. The cost figures established by the Chino company should be of interest, not only to mining men, but also to engineers and contractors who have to do with excavations of various kinds.

The total average cost for blasting, loading, and hauling per ton of ore for the period October, 1910, to September, 1914, was about 20 cents; of this, about 12 cents was for labor and about 8 cents for supplies. The lowest average monthly cost was 12.9 cents per ton, 7.8 cents of which was for labor and 5.1 cents for supplies. The highest average monthly cost was 60.5 cents for July, 1911; 43.5 cents of that cost was for supplies and 17 cents for labor. The purchase of an unusually large store of supplies, charged against this month, caused this abnormally high average.

The total average cost of stripping per cubic yard, from October, 1910, to August, 1914, was 31.5 cents, equivalent to 15.2 cents per ton. Of this, 20.1 cents was for labor and 11.4 cents for supplies. During the same period, the lowest monthly average cost per cubic yard was 23.4 cents, 15.2 cents of which was for labor and 2.8 cents for supplies. The highest monthly average cost was 36.8 cents per cubic yard, of which 24.3 cents was for labor and 12.5 cents for supplies.

During the year 1913, the average cost of mining and hauling to the mill and dumps all classes of material was 36.87 cents per cubic yard. The cost of handling waste alone was 33.43 cents per cubic yard in place, or a little over 16 cents per ton. The cost per ton for blasting, loading, and hauling the ore was 23.13 cents per ton. In general the cost per ton is greater for ore than for waste because of the limited areas in which ore is loaded and the consequently more intermittent character of the loading. Other delays arise from occasional shortage of standard railroad cars for loading ore. The breaking of large blocks of ore by blasting formerly added something to the ore cost. The new plant for crushing coarse material at the mine is intended to obviate such blasting.

MILLING.

About 10 miles southwest of the terminus of the railroad line at Santa Rita, it emerges from the narrow winding Whitewater Valley and gains the open plateau. Near here, with many miles of open country in which to expand, the mill town of Hurley was laid out, and the concentrating plant was built. The discussion following is not intended to be a complete description of this splendid modern mill, but it calls attention to certain details not mentioned in the account of the mining operations.

The ore is hauled from the mine to the mill by the Santa Fe Railroad Co. Each train, handled by one locomotive and composed of 25 to 27 50-ton steel cars, carries 1,250 to 1,350 tons of ore. At Hurley the cars are run out on a very high trestle over the top of the crude-ore bins, into which they discharge by gravity. The course of the ore through the crushing plant at the mill is shown by figures 17 and 18.

Description of parts shown in figure 17.

1. Fifty-ton side-dump ore cars. Weight of car, 44,700 pounds. Length, 35 feet.
2. Grizzlies of 60-pound rails over bins; openings, 12 by 13 inches.
3. Crude-ore bins. Total storage capacity, 14,500 tons; available storage capacity without poking, 5,000 tons; each bin 34 feet wide by 28 feet 6 inches deep by 300 feet long inside.
4. Forty feeders of the caterpillar type. Speed of apron, 10 feet per minute; capacity of each feeder, 1.25 tons per minute—75 tons per hour.
5. Two 30-inch conveyor belts moving 245 feet per minute; about 325 feet from center line to center line.

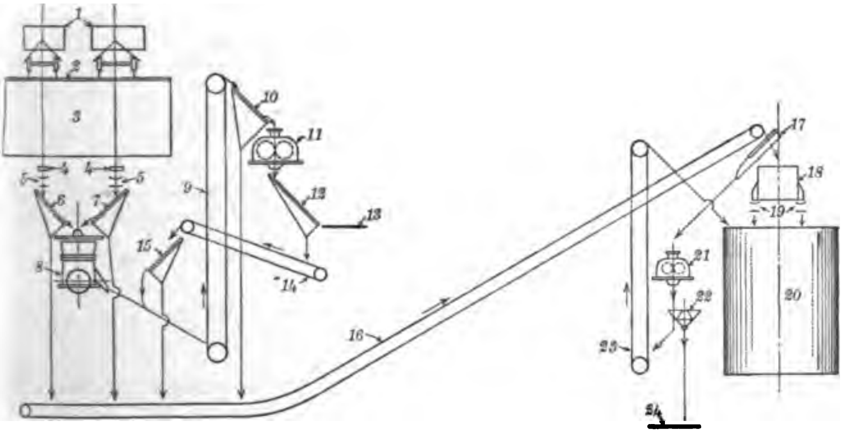


FIGURE 17.—Flow sheet of plant for crushing coarse material, Hurley mill.

6. Grizzly, 1 inch by 4 feet by 12 feet; bars ¾ inch by ½ inch by 3 inches.
7. Grizzly, 1 inch by 4 feet by 11 feet; bars ¾ inch by ½ inch by 3 inches.
8. No. 8 McNally crusher; 375 revolutions per minute; crushing to 3 inches maximum size.
9. Elevator; belt 36 inches, 12 ply; head pulley, 60 by 38 inches; boot pulley, 48 by 38 inches; 49 feet 4 inches from center line to center line; belt speed, 440 feet per minute; buckets, 17½ by 8 by 10 inches; projection, 18 inches between buckets.
10. Grizzly, 1 inch by 4 feet by 9 feet; bars, ¾ inch by ½ inch by 3 inches.
11. Rolls, 72 by 20 inches; peripheral speed of roll shells when new, 1,000 feet per minute.
12. Grizzly, 2½ inches by 3 feet by 8 feet 9 inches; for removing large pieces of native copper, wood, etc.
13. Steel platform at bottom of grizzly.
14. Twenty-inch conveyor belt; 20 feet from center line to center line; inclination, 20°; speed, 330 feet per minute.
15. Grizzly, 1 inch by 3 feet by 7 feet.
16. Conveyor belt, 36 inches wide; about 275 feet from center line to center line; maximum inclination, 20°; speed, 300 feet per minute.
17. Primary sampler; cuts sample every five minutes.
18. Receiving hopper.
19. Two 24-inch shuttle conveyors; 24-inch belts, 120 feet long; speed, 350 feet per minute.
20. Ten steel tanks, 24 feet 6 inches in diameter by 40 feet high.
21. Rolls, 14 by 27 inches.
22. Vezin sampler, cuts 20 per cent of (21) roll product.
23. Elevator, returning material rejected from samplers to bins for fine ore; 6-ply belt, 6 inches wide.
24. Floor on which sample is quartered by hand.

Description of parts shown in figure 18.

1. Fine-ore bins; two cylindrical steel tanks 24 feet 6 inches in diameter by 40 feet deep; approximate capacity 1,000 tons each.
2. Four feeders of the caterpillar type.
3. Two 20-inch belt conveyors, about 20 feet from center line to center line; speed, 250 feet per minute.
4. Two 24-inch wet elevators; 24-inch, 12-ply belts; speed, 400 feet per minute; about 62 feet from center line to center line; buckets staggered on belts; pitch of alternate buckets, 9 inches; buckets 12 by 7 by 8 inches.
5. Six 36-inch by 48-inch impact screens; 600 impacts per minute.
6. Two 42-inch by 16-inch rolls; peripheral speed of shells when new, 1,000 feet per minute.
7. Four 36-inch by 48-inch impact screens; 600 impacts per minute.
8. Two jigs with two 6-mesh screens each; screens 24 by 36 inches; 112 revolutions per minute; 1½-inch stroke.
9. One 30-inch wet elevator; 30-inch, 12-ply belt; speed, 450 feet per minute; 60 feet from center line to center line; buckets, 15 by 8 by 10 inches, staggered on belt; pitch of alternate buckets, 9 inches.
10. Four 4-spigot Richards-Janney classifiers.
11. Two desanding tanks, 5 feet 3 inches by 7 feet 2 inches by 6 feet 9 inches deep.
12. Chilean mill feed tank, 3 feet by 24 feet by 6 feet 6 inches deep.
13. Three Garfield Chilean mills, 32½ revolutions per minute.
14. Two three-spigot Richards-Janney classifiers.
15. Two three-spigot Richards-Janney classifiers.
16. Four five-spigot Richards-Janney classifiers.
17. Six 4-foot by 12-foot Garfield tables; 245 revolutions per minute; ¾-inch stroke.
18. Two 4-foot by 12-foot Garfield tables; 245 revolutions per minute; ¾-inch stroke.
19. Two 4-foot by 12-foot Garfield tables; 245 revolutions per minute; ¾-inch stroke.
20. Two 4-foot by 12-foot Garfield tables; 245 revolutions per minute; ¾-inch stroke.
21. Two 4-foot by 12-foot Garfield tables; 245 revolutions per minute; ¾-inch stroke.
22. Six No. 5 Wilfley tables; 245 revolutions per minute; ¾-inch stroke.
23. One double three-compartment jig; 188 revolutions per minute; 2-inch stroke; 26-inch by 38-inch screen; first and second compartments, 7-mesh, 0.035 brass wire; tail screen, 9-mesh, 0.035 brass wire.
24. Four Isbell vanners; corrugated belts, 6 by 10 feet; 2-inch side stroke; 130 revolutions per minute; 7½-inch slope.

25 and *26*. Each consists of four vanners same as *24*, but having 7-inch slope.

27. Four vanners same as *24*, but having 6½-inch slope.
28. Four vanners same as *24*, but having 6-inch slope.
29. Four vanners same as *24*, but having 7-inch slope.
30. Four vanners same as *24*, but having 6-inch slope.
31. Four vanners same as *24*, but having 6-inch slope.
32. Twelve vanners same as *24*, but having smooth belts; 120 revolutions per minute; 4½-inch slope.
33. Thirty-two vanners same as *32*.
34. Twenty-eight vanners same as *32*.

35, *36*, *37*, and *38*. Each consists of ten steel-cone tanks; 9 feet 5 inches in diameter; 60° slope.

39. Fifteen double wooden tanks; water-recovery plant, equivalent to 50 cone tanks or 10 cone tanks per section; serving five sections.
40. Two slime pumps.
41. Four pumps; 10-inch, special, class "B" volute; two sets of two each, series connected; serve five sections.
42. Two muddy-water sumps, serving five sections.
43. Four pumps; 10-inch discharge; special, class "B" volute, serving five sections.
44. Two sumps for all vanner concentrates, serving five sections.
45. Two 4-inch centrifugal pumps, series connected, taking material from sumps *44*, and serving five sections.
46. One 4-compartment Richards-Janney classifier.
47. One 4-foot by 12-foot Garfield table, modified.
48. One 4-foot by 12-foot Garfield table, modified.
49. One 4-foot by 12-foot Garfield table, modified.

50, *51*, and *52*. One vanner same as *24*, but having 6-inch slope.

53. Concentrate sump, serving five sections.
54. Three 6-inch centrifugal concentrate pumps, serving five sections.
55. One primary concentrate sampler, Vezin type, serving five sections.
56. One secondary concentrate sampler, Vezin type, serving five sections.
57. Sixteen concentrate bins, 22 feet 6 inches by 15 feet by 12 feet deep. All bins are served by a 4-ton Gantry crane with a 24 cubic foot clamshell bucket.
58. One tailings sampler, pendulum type, cutting the tailings stream once in 6 minutes; serves five sections.

NOTES.—*46* to *52* inclusive, constitute concentrate cleaning plant. Three such plants serve five sections.

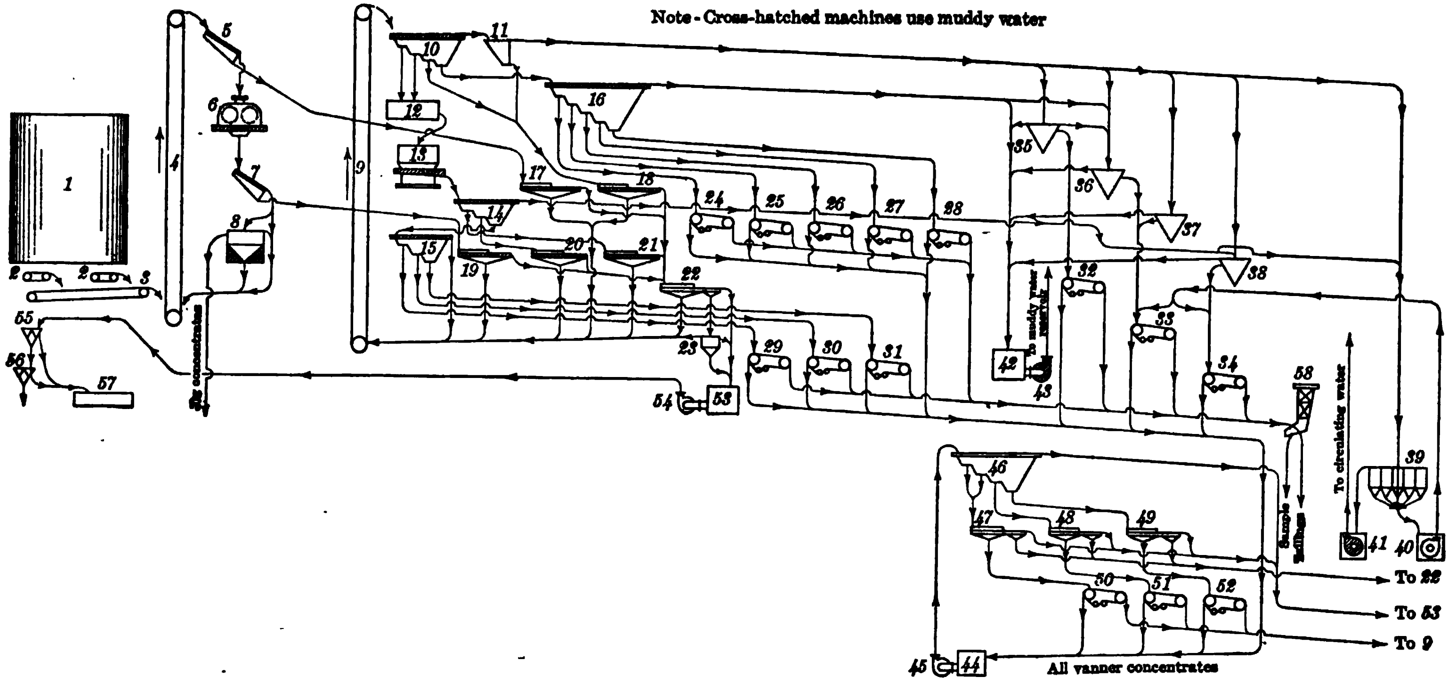


FIGURE 18.—Flow sheet of a section of operations at Hurley mill.

Since the installation of the crushing plant for coarse material at the mine, the grizzlies (*2*, fig. 17) across the top of the crude-ore bins, consisting of 60-pound rails giving openings 12 by 13 inches, are seldom needed. The capacity of the receiving bins *3* is 5,000 tons, and they discharge, through 40 caterpillar feeders, *4*, arranged along the bottom, 20 on a side. These supply two 30-inch conveyor belts, *5*, which carry the ore to the second set of grizzlies, *6* and *7*, from which the undersizes drop onto the main belt conveyor *16*, and the oversizes go into a No. 8 McNally crusher, *8*, breaking to a maximum size of 3 inches. From this the total product is raised by an elevator, *9*, and passes over a third grizzly, *10*, with openings ¾ by ½ inch by 3 inches. The oversize from this goes to rolls *11* and the undersize to the main belt conveyor *16*.

Another grizzly gets the product from the rolls *11*. This treatment is mostly to take out, as oversize, flattened pieces of native copper, wood, etc. The undersize goes to another elevator, *14*, then over another grizzly, *15*. The oversize from this is elevated and again passes through the rolls *11*, the undersize going to the main belt conveyor *16*. The latter elevates the crushed ore to a receiving hopper, *18*, which distributes it onto shuttle-belt conveyors, *19*. These discharge into one of 10 steel ore tanks, 24½ feet in diameter and 40 feet high. A primary sampler, *17*, cuts a sample from the crushed-ore stream every five minutes. This sample passes through rolls, *21*, and is then resampled by a Vezin sampler, *22*, the rejected material being returned to the fine-ore bins by an elevator, *23*. The sample retained is quartered by hand on a table, *24*.

From the fine-ore bins (*1*, fig. 18) caterpillar feeders, *2*, deliver the ore to belt conveyors, *3*. From them it is raised by an elevator, *4*, and delivered to impact screens *5*, with the oversizes going to rolls *6*, and the undersizes to Garfield tables *17*. The product from the rolls goes to other impact screens *7*, the fines from which go to Garfield tables *19*, and the oversizes to jigs *8*. The jig oversizes go back to the elevator *4*, and the concentrates finally reach the concentrate bins. From the Garfield tables the concentrates go to Wilfley tables *22*, and the tailings go back by elevator *9* to classifiers *10*. Of the five products from these, the two coarsest go to Chilean mills *12* and *13*, the finest to desanding tanks *11*, the next finest to fine classifiers *16*, and the intermediate to Garfield tables *18*. All the products from the fine classifiers *16* go to vanners *24* to *28*. The products from the Chilean mills *12* and *13* go to classifiers *14*, which give three products, the two coarser going to Garfield tables *20* and *21* and the finest to a fine classifier, *15*. The three products from the latter go to vanners *29* to *31*. The Wilfley tables *22* give three products, the coarse tailings going back by elevator *9* to classifiers *10*, the intermediate to jigs *23*, and the concentrates to concentrates sump *53* and thence to storage bins *57*. All the concentrates from vanners *24* to *31* go to sumps *44*, from which pumps *45* elevate them to the cleaning plant *46* to *52*. The tailings from the vanners go out onto the waste dump, being automatically sampled by samplers *58* on the way. The overflow from the desanding tanks *11* and from the classifier *16* goes to a series of steel-cone settling tanks *35* to *38*. From these the overflow goes to muddy-water reservoirs *42*, where it is somewhat clarified by settling, so as to be used over again. The slimy sediment from the cones goes to smooth-belt vanners *32* to *34*, which give tailings that go to the waste dump and concentrates that go to the cleaning plant *46* to *52*. This comprises a Janney classifier, modified Garfield tables, and vanners, designed to handle very fine material. The concentrates from the vanners of this cleaning plant are mixed with the concentrates from all the other vanners, and go through the cleaning plant again; the tailings go back to the classifiers *10*. The intermediate product from the modified Garfield tables of the cleaning plant goes back to Wilfley tables *22* and the concentrates from the latter go to the concentrates sump *53* and bins, as do the concentrates from the Wilfley tables and the overflow from the classifiers *46* of the cleaning plant.

The foregoing outline, in conjunction with figures 17 and 18, gives in brief form the chief operations of the mill. Since the time of the authors' visit in 1914 alterations and improvements have been added to the plant, but these are perhaps too technical to be treated in any but a report addressed to metallurgists.

The mill has a maximum capacity of almost 8,000 tons per 24 hours, when the character of the ore is favorable, but 6,000 tons is considered a fair daily average. To operate the total milling plant, consisting of five concentrating sections similar to that shown in figure 18, and the coarse-crusher section (fig. 17), about 3,500 horsepower of electrical energy is required.

The average extraction for 1913 was 67.31 per cent from 1,942,700 dry tons milled, equivalent to 5,322.4 tons per day. During a period of about five months a section of the mill was idle owing to modification in the scheme of treatment; hence the tonnage for the year does not represent full capacity. The ratio of concentration for the year was 10.61 to 1, with the concentrates averaging 14.518 per cent copper, or a recovery of 27.37 pounds per ton of ore. The ore treated for the year averaged 2.033 per cent copper. The ratio of concentration was lower than in 1914 because of the high iron content of certain of the ores mined during the year.

The ores treated during 1914 carried less iron and gave a better concentration. For the month of September, 1914, the ratio of concentration was 17.32 to 1, from 101,300 dry tons milled. The lessened tonnage treated was due to the war conditions. The actual recovery was 73.438 per cent of the copper content of the ore. The concentrates carried 27.67 per cent of copper. Any excess of silica over iron is penalized by the smelter. Toward the end of 1913 a concentrate recleaning plant was added to the mill and this helped to make a cleaner product, thereby lessening the freight and smelter charges on the copper actually produced.

HURLEY WATER SUPPLY.

A most important question which had to be solved before a site for the new mill could be decided on was the matter of an adequate water supply. Such a supply, it was found, could be obtained from springs, wells, and the subsurface flow of creeks within piping and pumping distance of the mill site at Hurley. An electrical pumping station on the "B Ranch" has a capacity of 750 gallons per minute. The pump is an 11-inch by 12-inch Aldrich triplex, driven by a 75-horsepower motor, which has to overcome a head of 250 feet. Similar pumping plants are on the Cameron and Whiskey Creeks. The Apache Tejo pumping station has two 14-inch by 18-inch quintuplex Aldrich pumps, each having a capacity of 2,500 gallons per minute; each is driven by a 400-horsepower electric motor, which has to overcome a total head of 400 feet. Only one of the pumps at the Apache Tejo station is operated at a time, the other being held in reserve. The supply of water for domestic use in the town of Hurley comes from this station.

Two reservoirs are in use at Hurley, one for clear and the other for muddy water. The clear-water reservoir gets the water from the pumping stations above described. It is 210 by 144 feet, and 12 feet deep. The muddy-water reservoirs get the settled water, already used in the mill, and caught and partly settled by the Whitewater No. 2 Dam. The size of this reservoir is about 60 by 70 feet and 12 feet deep. The muddy water is handled by six 10-inch, Class B, Worthington volute pumps, each having a capacity of 2,500 gallons per minute against a head of about 95 feet, and each driven by a 75-horsepower motor. The Whitewater No. 2 Dam and the settling reservoirs near it have a storage capacity of about 15,000,000 gallons. The water that flushes the tailings out of the mill is there ponded and settled to be used again. When the new spillway at the Whitewater No. 1 Dam is completed there will be a storage capacity for muddy and flood water of 500,000,000 gallons. The muddy water, after partial clarification by settling, is used over again on certain of the tables and classifiers. It may contain up to 4 or 5 per cent of solids.

The total amount of new water used in the mill during the month of July, 1914, was 52,569,637 gallons. With this about 560,000,000 gallons of muddy water was pumped to be used over again. The total ore treated during the month was 188,400 tons. The water used per ton of ore treated was 325.6 gallons of new water and 2,961.8 gallons of muddy water, a total of 3,287.4 gallons. The water used for domestic purposes in the town of Hurley during July amounted to 8,768,775 gallons.

MILLING COSTS.

The total average cost for all the ore treated in the mill up to September, 1914, was 58 cents per dry ton. In 1914, the lowest average cost for one month was 47 cents per dry ton, for June; during that month 201,000 dry tons was treated. The highest monthly average was 58 cents for the month of September, 1914, with only 101,300 tons milled. Since the mill opened, the total average cost of crushing coarse material (fig. 17) to a maximum size of 1¼ inches, has been 4 cents per dry ton, including supplies, operation repairs, remodeling, etc. During September, 1914, in spite of the lessened tonnage handled, the crushing of coarse material amounted to only 1.8 cents per dry ton. The crushing of fine material to a maximum size of 40 mesh, has cost, on the average, 11 cents per dry ton. For September, 1914, it averaged 9.7 cents. The concentration of the crushed material on the different tables, vanners, jigs, etc., has averaged about 13 cents per dry ton.

The total average milling expense, since the mill opened up to September, 1914, expressed in cents per dry ton of ore treated, is the resultant of the following charges: Light and power, 7.9 cents; water service, 4.8 cents; laboratory and sampling, 1.3 cents; Hurley office expense, 1 cent; engineering and surveying, 0.2 cent; tailings expense, ponding tailings, etc., 0.9 cent; mill heating, 0.1 cent; warehouse expense, 0.5 cent, making a total direct operating expense of 16.7 cents per dry ton of ore treated. A general mill-expense charge of 6 cents, probably covering experimental mill work, etc., together with 6 cents for administration and 1.4 cents for maintenance, raises the cost of mill-operation to 30.1 cents. This, when added to the cost of crushing coarse and fine material and concentrating, 4, 11, and 13 cents, respectively, makes the grand total of 58.1 cents per dry ton treated.

When the mill again begins to handle ore up to its full capacity, the recent adjustments and improvements made in it should considerably better these general average costs.

In connection with the Hurley plant there is a large assay shop where about 100 assays per day are made. Experimental work on the ores is also carried on in a well-equipped laboratory, with the object of still further reducing the cost and increasing the efficiency of concentration.

REPAIR SHOPS AT HURLEY.

At Hurley there are also storehouses and extensive machine and repair shops. The completeness of the latter is indicated by the following list of equipment:

Equipment in Chino company shops at Hurley.

Equipment in mechanical department.

One Woodward & Powell, jr., patented 42-inch by 42-inch by 12-foot planer.
One J. G. Blount No. 5 double-head emery grinder.
One New Haven 50-inch by 24-foot triple-geared, screw-cutting engine lathe.
One Prentiss 24-inch by 22-foot engine lathe.
One Davis 12-inch by 8-foot quick-change gear lathe.
One Steptoe 20-inch back-geared crank shaper complete.
One Blount 20-inch wet tool grinder, complete, with 20-inch by 2½-inch emery wheels.
One Yankee twist-drill grinder.
One Williams No. 5 12-inch pipe machine.
One Niles 5-foot semiuniversal radial drill.
One Hoefer 21-inch vertical drill press.
One Owen No. 2 milling machine.
One Acme 2-inch single-end bolt-threading and nut-tapping machine, class "A."
One Cleveland Punch and Shear Co. No. 0 plate-bending rolls.
One Robertson No. 2 rapid power hack saw.
One Ryerson 1,200 pound single-frame steam hammer.
One American 36-inch lathe, with 18-inch bed, double back-geared.
One American No. 3 high-speed radial drill.
One Higley No. 12 cold metal saw.
One Ryerson-type, radial drill, with 8-foot arm.
One Ryerson & Son Cleveland universal splitting shear.
One American Tool Works 18-inch, new-pattern, heavy-duty engine lathe.
One Cleveland Punch & Shear Co., style C, solid-frame single end punch-and-shearing machine.
One Acme 1-inch single-bolt cutter, class "A."
One Green River No. 55 opening die bolt cutter.
One Racine high-speed power hack saw, 6-inch capacity.
One Brown & Sharpe cutter grinding machine No. 2.
One Lodge & Shipley screw-cutting heavy-duty engine lathe.
One Racine high-speed draw-cut hack saw, 6-inch capacity.

Carpenter-shop equipment.

One power mortising machine.
One 24-inch single-cylinder planer and matcher.
One 24-inch hand planer and jointer.
One 16-inch variety saw.
One Universal 16-inch woodworker's saw.
One single-spindle vertical boring machine.
One 36-inch band saw.
One pattern maker's wood lathe, 24-inch swing.
One Buffalo knife grinder.
One Universal No. 8–f trimmer.
One double emery-wheel stand.
One 36-inch grindstone with iron frame.
One 24-inch rip saw.
One cut-off saw, 36-inch swing.

During the month of June, 1914, 33 mechanics and helpers were employed in the mechanical department, and 8 carpenters and helpers in the carpenter shop of the Hurley shops.

During the month of June the total cost of repairs at Hurley, including labor, supplies, and overhead charges, was 1.96 cents per dry ton of ore milled. The cost for September, 1914, was 1.89 cents, and for April, 1915, 1.32 cents per dry ton milled.

GENERAL ENGINEERING DETAILS.

Where operations are so extensive as those at Santa Rita, necessarily a wide range of engineering problems is presented for solution. Not only is much field work and mapping necessary, but a great deal of yardage measuring and computing must also be done each month. Railroad-engineering problems are common, and jobs in machine design, architecture, reinforced concrete work, hydraulics, etc., make calls on the engineering staff from time to time. This staff embraces a chief engineer and five to eight assistants.

MONTHLY ESTIMATES AND STATEMENTS.

At the end of every month stripped and mined areas are measured, and the yards and tonnage are computed. The measurements are made by stadia, and the information is plotted on a coordinated and contoured working map, which has a scale of 30 feet to the inch. The stadia stations are accurately located by transit from triangulation points. A field party consists of a transitman, a recorder, and two rodmen. The field notes are recorded in the transit book in the following form:

Specimen record in transit book.

Date ....

Low Line No. 8 Dump.

Party: ...., Transitman; ...., Recorder; ...., Rodman (top).

..........., Rodman (bottom).

[Field page.] [Office page.]

Station.[a]	B. S.[b]	Horizontal angle.[c]	Stadia distance.[d]	Vertical angle.[e]	High or low sights.[f]	Horizontal distance.[g]	Vertical distance.[g]	Elevation.[h]
12 (H. I. 4.9); elevation	△ N	352°00′	408	+3°26′	....	408	+24.5	6370.4
6,345.9..................	....	355°15′	388	+4°45′	+2ft.(B.L.)	386	+32.1	6376.0
	....	358°15′	370	+3°43′	....	370	+24.0	6369.9

[a] Transit station.
[b] Backsight (on triangulation station N).
[c] Read to nearest 15 minutes.
[d] Stadia distance read to the nearest foot.
[e] Read to nearest minute.
[f] High or low sights read (called "boot leg").
[g] Actual, obtained from stadia reduction tables.
[h] True elevation of ground calculated.

When information similar to that contained in the specimen statement is put on the map, a color distinctive from that used the previous month is employed. Sections across the entire cut or dump are taken every 25 feet and are plotted as shown in figures 19 and 20.

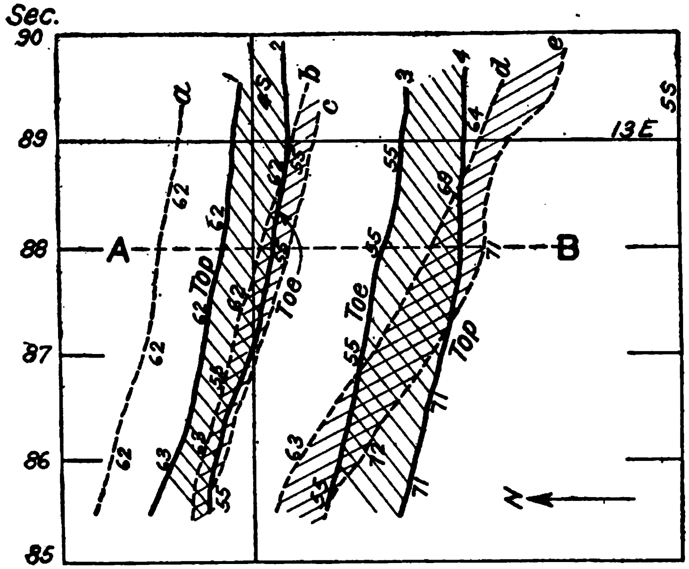


Figure 19.—Plan map of excavation and of filling during one month. The dotted lines *a* to *e* are each drawn through points having approximately the same elevation, on surface of previous month's excavation; solid lines *1* to *4* show points having same elevation on present surface. Elevation is indicated in feet. The figures *85* to *90* refer to sections on plotting map; *5S*, 13*E*, refer to coordinates of drill holes shown on engineer's records of yardage of excavation.

The surface as measured the previous month is shown on the cross section (fig. 20) by dotted lines, and the more recent surface is shown by solid lines. A plan map of excavation and filling for one month is shown in figure 19, which represents an area of which figure 20 shows a cross section.

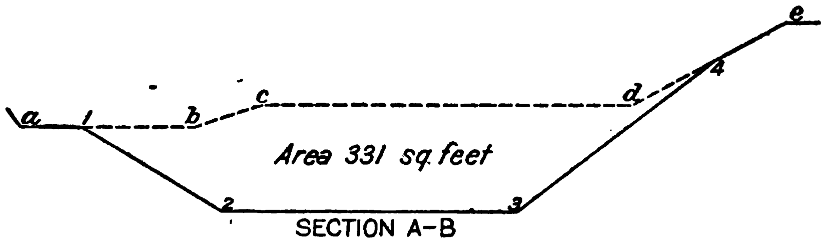


Figure 20.—Plotted cross section of month's excavation. Area represented is that of a vertical cross section through A B, in figure 19. The dotted line represents surface as measured in previous month.

The end areas are scaled directly off the cross sections, and the average of the two end areas, multiplied by the distance between the cross sections, gives the cubical contents of each excavated block.

The plotting and computations are checked, and then the results are entered in the "Steam-Shovel Computation Book" in the following manner:

Specimen entries in "Steam-shovel Computation Book."

Section No.	Total cut.[a]		Ore estimate.		
	Area.	Volume.	Area.	Average end area.	Volume.
(Wedge, 15 feet) to 95	*Sq.ft.* (Noted if any.)	*Cu.ft.* (Noted if any.)	*Sq.ft.* 14	*Sq.ft.* 7	*Cu.ft.* 105
95 94			14 56		
			70	35	875
94 93			56 174		
			230	115	2,875
93 92			174 284		
			458	229	5,725

[a] Stripping and mining.

Under the column headed "Section No." is recorded the wedge of ground excavated during the preceding month between cross sections 96 and 95. Under "Total cut" is shown the total excavation of waste and ore; under "Area" the areas of the cross sections in square feet are given, cross section 95 had an area of 14 square feet, and cross section 94 one of 56 square feet. The average area of the two sections, 35 square feet, is given under the column designated "Average end area." Multiplying this by 25 feet, the distance between the two cross sections, gives 875 cubic feet, the volume of the prism between sections 94 and 95, removed during the month. In like manner the cubical contents of all the other prisms are calculated. In the calculations 13 cubic feet of rock in place, and 21 cubic feet of broken rock, are used as the equivalent of a ton of 2,000 pounds. Additions to and subtractions from ore and waste dumps are calculated in the same manner.

The records of excavation for each bench are reported separately, as are the records of the excavation work of each steam shovel. If a shovel excavates from two or more benches during the month, the proportion moved from each is determined from the bench and the shovel records, and the proper apportionment made. The assay records and the number of cars sent to the crusher, direct to the mill, or direct to the smelter, determine the amount of the material classed as ore. The number of cars sent to the waste dumps determines the amount of the material excavated classed as waste. Of course, these data are checked against the engineers' estimates of solid cubic yards moved, etc.

A monthly comparative statement of the work done by each shovel is compiled for each month. This table also shows the number of cars of material loaded by each steam shovel and the capacity of each car. Records are kept by each steam-shovel foreman of the number and capacity of cars loaded and whether they contain ore or waste. The table following shows the results of steam-shovel operations during July, 1914:

Results of steam-shovel operations at Santa Rita during July, 1914.

Steam shovel No.	Solid cubic yards of waste handled.[a]	Tons of ore handled.[b]	Material in place handled, solid cubic yards.[a]	Shifts worked.[c]	Average solid yards per day (two shifts).
1	14,222	40,415	33,681	50.5	1,334
2	57,437	....	57,437	56.2	2,485
3	44,243	26,665	57,082	56.0	2,482
4	74,469	....	74,469	55.9	2,664
5	57,648	2,451	58,828	49.7	2,367
6	28,953	8,909	33,242	45.5	1,461
7	20,590	38,305	39,033	56.8	1,668
8	47,356	1,466	48,062	51.6	1,863
9	11,691	108,010	63,696	57.1	2,231
10	....	4,862	2,341	11.2	418
Total	356,609	231,083	467,871	488.5	1,916

[a] Compiled from engineers' cross-section estimates.
[b] Taken from mill returns.
[c] From steam-shovel foreman's report.

A table showing the broken-rock yardage capacity of all the different ore cars used is a convenience in making estimates. It shows, for instance, that a car having a capacity of 100,000 pounds can carry 38 cubic yards; a car having a capacity of 80,000 pounds is loaded with 30 cubic yards; a car having a capacity of 60,000 pounds carries 23 cubic yards; and a car of 55,000-pound capacity contains 21 cubic yards of broken rock. For convenience of calculation this table is extended so that one can see at a glance that a train of, say, 50 cars of 100,000-pound capacity can carry 1,900 cubic yards of ore.

The engineering department also posts a special form that serves as a daily indicator of steam-shovel operations for the information of the manager and superintendents. A specimen form follows:

Specimen report of day's steam-shovel operations, for information of mine manager and superintendent.

Date.	Shift (D=day N=night).	Accumulative car yards per shift.[a]	Total car yards per day.[a]	Accumulative car yards per day.[a]	Accumulative for month.[b]	Comparison with previous month.[c]	Remarks.
8	D	480	480	480	622	x 142	Shovel No. — shut down for repairs.
9	D	1,056	576	1,056	622	+ 434	

[a] From individual shovel records.
[b] From previous month's record.
[c] Shows gain or loss as indicated by difference between figures in two preceding columns. x indicates car-yard shortage, as compared with the previous month; + indicates car-yard increase.

The engineering computations in the office are greatly facilitated by detailed and comprehensive tables and diagrams, prepared for reducing the stadia observations, giving elevations, horizontal distances, etc.

On a plain coordinated wooden surface, about 4½ by 6½ feet, the drill holes are accurately located, and each is shown as a ⅛-inch hole bored into the wood. In these holes ⅛-inch round sticks are placed. The height of the stick above the surface represents the depth of the hole. On each stick, the ore body or bodies that the hole cuts are painted, according to scale, showing the proper thickness and spacing. This gave a fairly good chart, or model, of the ore bodies, showing their depth, thickness, etc. However, this chart proved much less useful than the cross sections of the ore bodies prepared from the drill-hole data.

MONTHLY, QUARTERLY, AND ANNUAL STATEMENTS BY ENGINEERING DEPARTMENT.

Monthly, quarterly, and annual statements and tables are compiled by the engineering department to give the following information: The amount of ore excavated, milled, and smelted; the total waste excavated and hauled to the dumps; the yardage of ore and waste taken from each bench; the costs of the various operations; the amount of ore remaining; its content of copper and value under given conditions, and other such data. In order to illustrate the manner in which these data are tabulated the following self-explanatory forms are given:

ORE AND STRIPPING.

Steam-shovel mining .... ore body.

SANTA RITA, N. MEX., ...

For month of ....

Bench.	Solid rock, cubic yards in place.	Earth and dumps, cubic yards in place.	Total cubic yards.	Cubic yards of stripping.	Per cent of copper in stripping.	Tons of ore.	Per cent of copper in ore.	Pounds of copper.

Remarks:

[This form is amplified to contain accumulative records of totals to date from Jan. 1.]

....

STEAM-SHOVEL OPERATION.

SANTA RITA, N. MEX., ...

For .... quarter, 19....

Steam shovel No.	Cubic yards of waste handled.	Tons of ore handled.	Yards in place handled.	Shifts worked.	Average solid yards per day (twoshifts).

Remarks:

....

TOTAL STEAM-SHOVEL WORK.

SANTA RITA, N. MEX., ...

For month of ....

Ore body.	Solid rock, cubic yards in place.	Earth and dumps, cubic yards in place.	Total cubic yards.	Cubic yards of stripping.	Per cent of copper in stripping.	Tons of ore.	Per cent of copper in ore.	Pounds of copper.

Remarks:

[This form is amplified to contain accumulative records of totals to date from Jan. 1. A form with similar headings is used for quarterly reports.]

....

WASTE DUMPS.

SANTA RITA, N. MEX., ...

For month of ....

Dump.	Cubic yards.	Tons.	Per cent of copper.	Pounds of copper.	Cars.	Tons per car.	Cubic yards per car.	Totals to date from January 1.			
								Cubic yards.	Tons.	Per cent of copper.	Pounds of copper.

Remarks:

....

ORE PILE.

Santa Rita, N. Mex., ...

For month of ....

Pile.	Cubic yards.	Tons.	Per cent of copper.	Pounds of copper.	Cars.	Tons per car.	Cubic yards per car.	Totals to date from January 1.			
								Cubic yards.	Tons.	Per cent of copper.	Pounds of copper.

Remarks:

....

MILL SHIPMENTS.

Santa Rita, N. Mex., ...

For month of ....

Ore from—	Shipping weight, tons.	Per cent of moisture.	Dry weight, tons.	Per cent of of copper.	Pounds of copper.	Cars.	Tons per car.	Totals to date from January 1.				
								Shipping weight, tons.	Per cent of moisture.	Dry weight, tons.	Per cent of copper.	Pounds of copper

Remarks:

....

SMELTER SHIPMENTS.

Santa Rita, N. Mex., ...

For month of ....

Ore from—	Shipping weight, tons.	Per cent of moisture.	Dry weight, tons.	Per cent of of copper.	Pounds of copper.	Cars.	Tons per car.	Totals to date from January 1.				
								Shipping weight, tons.	Per cent of moisture.	Dry weight, tons.	Per cent of copper.	Pounds of copper

Remarks:

....

ORE AND WASTE DUMPS.

MILL SHIPMENTS.

SANTA RITA, N. MEX., ...

For .... quarter, 19...

Ore dumps.					Waste dumps.				
Dump.	Cubic yards.	Tons.	Per cent of copper.	Pounds of copper.	Dump.	Cubic yards.	Tons.	Per cent of copper.	Pounds of copper.

SMELTER SHIPMENTS.

Ore from—	Shipping weight, tons.	Per cent of moisture.	Dry weight, tons.	Per cent of copper.	Pounds of copper.	Cars.	Tons per car.

Remarks:

....

HEALTH AND SANITARY MEASURES.

HOSPITAL AT SANTA RITA.

When the new company acquired the Santa Rita property and began operations on an extensive scale, a suitable hospital for their employees seemed highly desirable. Consequently, a well-ventilated one-story frame structure with wide open porches was built and adequately equipped. The hospital has been in existence four years, and in that time with a staff of two doctors and four nurses, it has successfully treated 1,314 cases. It is maintained by fees of $1.50 per month from all single employees and $1.75 from all married employees. The fees entitle the employees and their dependents to hospital services, medicine, and treatment during illness. Separate charges are made, however, for childbirth cases and also for cases of illness resulting from drunkenness or fighting, and for treatment of venereal diseases. Patients not entitled to free treatment give $2 a day in addition to nominal charges for surgical operations.

The following data regarding cases treated during the two years ended February 15, 1913, show how useful and successful the hospital has been:

Data regarding cases treated in company hospital during two years ended Feb. 15, 1913.

Cases in hospital	665
Deaths	21
General operations	106
Abdominal operations	32
Deaths following operations	[a] 2
Pneumonia cases	34
Deaths from pneumonia	5
Accident cases	157
Deaths from accident	2
Typhoid cases	37
Deaths from typhoid	1
Obstetrical cases	35
Deaths from obstetrical cases	0

CAUSES OF DEATHS.

Tuberculosis	2
Organic heart disease	1
Pneumonia	5
Cancer	1
Enterocolitis	2
Hookworm	1
Following injuries	2
Meningitis	1
Typhoid	1
Cerebral hemorrhage	2
Gunshot wound	1
Peritonitis	1
Syphilis	1
Total	[b] 21

ACCIDENT REPORTS.

When an accident occurs to an employee his foreman at once issues a report on the following form to the general office:

NOTICE OF AN ACCIDENT TO AN EMPLOYEE.

Name of person making report.................... Occupation................
.....................Street. ....City. ....State.
Date and hour of accident.......................................191.., ...m.
Date of this report.................... 191...
Injured person:
Name............................ Address............................
Age............... Family............... Weekly wages, $............
Occupation...
How long employed in this work?......................................
How long employed prior to accident?................................
General duties...

---

[a] One was advanced appendicitis and one was gunshot wound of stomach, liver, and intestines.
[b] Of these, 7 did not live 2 hours after admission to hospital.

The injury:

Nature and extent...

Was surgical aid rendered?.......... By whom?.........................

Taken home or to hospital?.......... Probable length of disability..........

Has injured returned to work?.......................................

The machine or appliance causing the accident:

What was it?...

State condition..................... When last inspected?.................

Whose control at time of accident?.....................................

Was there any defect in the machine or appliance? State fully: ....

....

Was light good?..................... Have broken parts (if any) been preserved?...

The accident:

Place?...

Was accident due to carelessness of injured or negligence of fellow workmen? If so, of whom?..

Statement of injured..

Name and address of foreman in charge of work.........................

Names and addresses of witnesses: ....

DESCRIPTION OF THE ACCIDENT.

(If necessary, to show cause of accident, draw rough sketch on back hereof.)

[Space for description.]

This notice made out by: ....

(State occupation.)

When the injured person has recovered, the following form certificate is executed:

Form for certificate of injury.

Claim No. ....

LIABILITY DEPARTMENT.

SURGEON'S REPORT AND CERTIFICATE OF CLAIM.

1. Name of injured...................... Occupation...........................
2. When first seen after accident..
3. Precise nature, location, extent of injuries and how received.................
4. Were such injuries the direct result of said accident.........................
5. I attended claimant from .... to ...., 19...., during which time the injuries above described constituted the sole and only cause of disablement; and I certify that he was totally incapable of following his usual occupation, because of such injuries, from the .... day of ...., 19...., to and including the .... day of ...., 19.....

....

Surgeon.

CERTIFICATE OF EMPLOYER, SUPERINTENDENT, OR TIMEKEEPER.

This is to certify that .... herein referred to, is insured under policy .... and was in my employment at a remuneration of .... per hour; that he was injured at the time and in the manner stated and, in consequence thereof, sustained continuous and total loss of time for the period of .... hours, amounting to $...........

Date ....

....

(Position.)

RELEASE.

Received of ....the sum of .... ($..............) dollars, in full satisfaction and discharge of any and all claims I have or may have against said .... because of a certain injury or injuries resulting from an accident sustained by me on or about the .... day of ...., 19...., while in the employ of the said ...., and further for any benefits that may be accrued to me by reason of said accident.

....
(Claimant.)

Date ....
Witness:
....
....

The number and the nature of accidents at the Santa Rita mine and at the Hurley plant for the years 1912 and 1913 are given in the following table:

Data regarding accidents at Santa Rita mine and Hurley plant, 1912, 1913.

	Year.	Killed.	Per cent.	Seriously injured.	Per cent.	Slightly injured.	Per cent.	Average number of employees per day worked.
Santa Rita mine......	1912	3	0.05	20	0.30	117	1.75	553
	1913	12	.11	25	.24	234	2.21	890
Hurley mill..........	1912	3	.036	15	.20	123	1.50	697
	1913	1	.01	5	.05	104	1.09	797

SANITARY EQUIPMENT.

A large number of the new company's houses are provided with modern plumbing. The sewage from these houses is discharged into cesspools. The smaller houses of the laborers are on large sloping lots and the waste water from the kitchens, baths, etc., is run out into the sand and gravel of the sloping surface and absorbed. Privies for these houses are placed at the rear of spacious lots, usually 100 feet away from the house. They are fitted with metal receptacles, which are removed periodically, and the contents thrown on the waste dumps and covered by carloads of waste. As a part of his general duties, the company's peace officer under the supervision of the chief physician maintains a vigilant watch over the sanitary condition of the camp.

RESCUE AND FIRST-AID WORK.

The Santa Rita main office has provided a suitable locker section in which three Draeger apparatus are stored. The equipment also includes duplicate oxygen bottles with water gage and liter bag. Hand flash lights are used exclusively.

Five employees of the company have been trained by Bureau of Mines field men, and received the bureau's certificates during the early spring of 1914; these five men have had periodic practice drills. It is the intention to train at least 10 more men in the use of the breathing apparatus.

The headquarters of the camp physician and his assistant is the company hospital, and on account of its close proximity to the mining operations no first-aid teams have been organized. The physician delivers periodic lectures on sanitation and elementary first-aid methods.

RECREATION.

The company officials encourage and contribute to the support of suitable forms of amusement for the employees and their families. Baseball and basketball teams are organized and participate in games arranged with similar teams from Hurley and Silver City. A rifle team enjoys the privileges of such teams as come up to the standards in the United States Army Regulations, obtaining healthful and instructive diversion. Two or three annual picnics give delightful outings to the men, women, and children of the camp.

TIME KEEPING, ACCOUNTING, AND WAREHOUSE METHODS.

A good working organization is one of the fundamentals of success in any business. A few years ago this factor was largely ignored by mining companies, but to-day it finds place in most of those that are successful. It, therefore, seems well worth while to call attention, in a general way, to the efficient system of time keeping, accounting, and warehouse methods in use at Santa Rita.

TIME KEEPING.

When skilled laborers are employed by the company, they are asked to fill out Form 10 so that some idea may be had of their responsibility and past achievements. From an unskilled laborer, no references are required other than a statement as to his last employment. Each foreman, on hiring any one, turns into the office a slip (Form 11). If the person employed is a skilled laborer, this form is yellow in color, and has the notice: "Bearer to furnish references." Each employee is given a number and is furnished with a cardboard check, 1½ by 2 inches, bearing that number.

FORM 10.—*Form filled in by applicant for position as skilled laborer.*[a]

APPLICATION.

Date..............................191...

MINING Co.,
Santa Rita, New Mexico.

GENTLEMEN: I hereby make application for work as.......................... and refer you to .... for my previous
(Give name of company and foreman, also address.)
record, and I hereby authorize and request .... to furnish same, including the cause of leaving .... service, if known. And I hereby release said .... from all liability for any damage whatsoever on account of furnishing such record.

....

Witness: ....

The above applicant .... was employed by the undersigned as .... from ....
to ....
Cause of leaving ....
Ability ....
Habits ....
Services were ....satisfactory.
Remarks: ....
Dated: .... Signed: ....

FORM 11.—*Form used in notifying timekeeper of the hiring of a new employee.*[b]

..............................191..

TO TIMEKEEPER:
I have hired ....
as a .... at ....
per day.

....
Foreman.

At the time office, which is conveniently situated, each employee, except the office force, the locomotive engineers, the firemen, and the steam-shovel crews, check in at the beginning of every shift, by calling out his number to the clerk at the window of the time office. The clerk puts opposite each man's number a horizontal black mark if the man is on the day shift and a red if on the night shift. The numbers are arranged on a daily pay-roll balance sheet, a section of which is shown as Form 12. The next check on the working time of the employees is that obtained by the time inspector, who goes around checking up the men found on the work. Forms 13*a*, 13*b*, and 13*c* indicate the character of the records kept in the time inspector's note book. These are left at the timekeeper's office to be checked with a perpendicular mark against each man's number on the daily pay-roll balance sheet. After the morning horizontal mark and the time inspector's perpendicular mark, each workman checked in has a plus (+) sign after his name on the sheet.

---

[a] Size of form used by operating company, 8½ by 11 inches.

[b] Size of original form 3¼ by 4¼ inches.

FORM 12.—*Register kept at time office.*[a]

DAILY PAY-ROLL BALANCE SHEET.

[Check men on shift with horizontal line in check column. Use black ink for day shift and red ink for night shift. Cross check from timekeeper's taken on the works. Enter hours and amount from work slip turned in at the end of each shift.]

0	Check.	Hours.	Amount.		1	Check.	Hours.	Amount.	
1									
2									
3									
4									
5									
6									
7									
8									
9									
10									

[a] Form used by operating company, 19¼ inches high by 36¼ inches wide. Provision made for 1,500 names on a sheet, 100 names in a column. Horizontal blue lines separate each number, every tenth number being followed by a purple line.

FORM 13*a*.—*Form used by time inspector for recording "time" of miscellaneous employees.*[a]

MISCELLANEOUS LABOR.

..............................., 191..

Day Night	No.	Hrs.	Remarks.
Car trimmers			
Switch tenders			
Switch tenders			
Flagman			
Water boys			

[a] Size of original form, 4¼ inches wide by 7¼ inches high, with three punch holes on left margin for binding.

FORM 13*b*.—*Form used by time inspector for recording "time" of steam-shovel crews.*[a]

SHOVEL NO. ....

Ore..........Hrs.
Strip.........Hrs.

..........................., 191..

Day Night	No.	Hrs.	No.	Hrs.
Engineer				
Fireman				
Craneman				
Coalman				
Pitmen				
Dumpmen				

[a] Size of original form, 4¼ inches wide by 7¼ inches high, with three punch holes on left margin for binding.

FORM 13c.—*Form used by time inspector for recording "time" of locomotive crews.*[a]

.........................., 191..

ACCOUNT:

		No.	Hrs.	No.	Hrs.
Foreman Eng. No.	[b] E				
	F				
	B				
Eng. No.	E				
	F				
	B				

---

[a] Size of original form, 4¼ inches wide by 7¾ inches high, with three punch holes on left margin for binding. Original form provides for record of 11 engines instead of 2, as shown.

[b] E, engineer; F, fireman; B, brakeman.

For the men engaged on special jobs that are to be charged to certain accounts, and also as an additional check on working time, Form 14 is used. This form is issued in different colors for the different occupations, and the occupation and the rate of pay of the employee are entered on each. For special jobs by skilled laborers, chargeable to certain accounts, Form 15 is used. If a special gang is engaged on work chargeable to different accounts, the distribution of the charge against each account is entered on Form 16.

All these forms, by keeping track of the proper distribution of charges, greatly facilitate bookkeeping. Each man's time record is kept in a large loose-leaf book composed of pages similar to that indicated by Form 17. At the end of the month this is turned in to the main office.

When an employee is discharged or quits the company's service, he is given a slip (Form 18) by his foreman. On the back of this his store, board, and rent bills are accounted for, or marked "none." The indorsements are made in ink and are signed by the officers responsible for them. The timekeeper then fills out Form 19, puts it in a sealed envelope, and hands it to the employee, who takes it to the cashier and receives his pay.

In common with most large employers, the mining company keeps personal record cards for each employee; the scope of these is shown by Form 20.

FORM 14.—*Form used in recording "time" of men employed on special jobs, the expense of which is to be charged to certain accounts.*[a]

Occupation Rate

....

Date..

P. R. No............ Hrs. Worked.................... Amount $.................

Charge to...

....

....

Foreman..................... ....

Hrs.
......
......
......

PUNCH HERE HOURS WORKED

1	14
2	15
3	16
4	17
5	18
6	19
7	20
8	21
9	22
10	23
11	24
12	25
13	26

FORM 15.—*Form used in connection with special jobs by skilled laborers, chargeable to certain accounts.*[b]

CARPENTERS.

SANTA RITA MINE,
Date.........................191..

Name.. Check No..................

Give full description of work done.	Hours.	Rate.	Amount.
Total			

Examined and approved:

....
Foreman.

---

[a] Size of original form, 3 by 5 inches.
[b] Size of original form, 4½ inches high by 7 inches long, on heavy flexible stock.

FORM 16.—*Form used in connection with work done by a special gang and chargeable against each of several accounts.*[a]

SANTA RITA MINE,
.................., *191* .

Occupation.	Number.	Hours.	Rate.		Amount.	

Charge to:

....

Examined and approved:

....
Foreman.

FORM 17.—*Form used in time office for keeping each man's time for a month.*[b]

TIME RECORD.

Name.................................... No............................

Occupation.	1	2	3	29	30	31	Total time.	Rate.	Amount.

Remarks. Total.

Deductions		
	Doctors and hospital	$
	Insurance	$
	Store	$
	Board	$
	Rent	$
	Check No.	$ Bal. due

FORM 18.—*Slip given by foreman to employee who is discharged or quits.*[c]

SANTA RITA, N. MEX., ..., 191 .

TIMEKEEPER:

Give bearer..

(No. ....) his time for services to date.

Quit
Discharged
No work
} Mark with an X.

Signed..

IMPORTANT.

Get this cleared at Santa Rita store, Santa Rita boarding house, and bunk house, then present at the office for payment between the hours of 1 p. m. and 3 p. m. daily except Sunday.

(Over.)

---

[a] Size of original form, 4 inches wide by 7 inches long, on heavy flexible stock.

[b] Original form is 12½ inches high by 14¼ inches long. Space for five names instead of one is provided, and columns for each day of the month. Each sheet is kept in time office until the end of the month, when it is turned in to the main office for balancing and checking.

[c] Size of original form, 3 by 5 inches.

[On reverse side.]

	Amount.		Signed.
Store Board Rent			

NOTICE.—The above must be filled in with ink; if there is no amount write "None" and sign name.

FORM 19.—*Slip handed by timekeeper to employee who leaves during month.*[a]

SANTA RITA MINE,
........................191....

Name.. P. R. No. ....

		Amount.
..........shifts, @ $..........		$.......
..........shifts, @ $..........		....
..........shifts, @ $..........		....
..........shifts, @ $..........		....
Total amount earned..		....
DEDUCTIONS.		
Doctor and hospital..	$.........	
Insurance..	....	
Store..	....	
Board..	....	
Rent..	....	
....	....	
....	....	
Total deductions..		....
Balance due..		....

The above statement is correct.

Signed..

FORM 20.—*Personal record card.*[b]

Name　　　Date employed　　　No.
Address　　　Boards
Dependents
Age　　Weight　　Height　　Eyes　　Hair　　M. or S.
Nationality　　　Engaged by
Last employer
Education

Date Dept. Occ. Rate Date Dept. Occ. Rate			

Left employ—Date　　　Reason:

---

[a] Size of original form, 5½ by 6 inches.

[b] Original card, ruled cardboard, 6 inches wide by 4 inches high.

ACCOUNTING.

When the labor-distribution cards (Form 15) have been signed and punched by the foreman and handed in to the main office they are entered on loose sheets similar to Form 21, which are kept in binders, forming large loose-leaf books. The labor data are all grouped, so that one or more of these sheets is used for firemen of all kinds, one or more for engineers, one or more for drillers, and so on. Each fireman, engineer, driller, etc., is charged against the particular locomotive or steam shovel fired by him for each day of his service during the month. These forms thus filled out present a complete daily record of labor charges classified and distributed against certain accounts. When this daily record has been summed up at the end of the month, the totals from it are entered on the Santa Rita labor-distribution sheets, the character of which is indicated in Form 22. This shows the total labor charges for the month properly distributed and arranged. One or more of these sheets is used for accounts that may be grouped under the general term, "Operation." In this group the columns are headed: Steam shovel 1, etc.; locomotives 1, etc.; drill 1, etc., and dump cars. The next group of headings on these sheets cover the following accounts: Rents and repairs, light service, automobile expense, prospect-drill expense, hospital fund, special water investigation. These are general ledger accounts and are carried in the general office at Hurley.

FORM 21.—*Form used in recording data on labor-distribution cards.*[a]

Month....................191..   Account....................

Date.	Shifts.	Amount.	Shifts.	Amount.	Total amount.
1					
2					
3					
4					
5					
6					
29					
30					
31					
Total					

---

[a] Original form 11 inches high by 26 inches wide, with wide margin on left for binding; 20 instead of 2 of the "Shifts-Amount" columns appear on the original, and space is provided for 31 days of the month.

FORM 22.—*Form used in recording total labor charges for each month, showing distribution.*[a]

SANTA RITA LABOR DISTRIBUTION.

Sheet No.......... ....191..

	Occupation.					
1	Assayers and samplers					1
2	Boilermakers and helpers					2
3	Brakemen					3
4	Blacksmiths and helpers					4
5	Blasters					5
6	Compressormen					6
7	Chauffeur					7
8	Car repairers					8
9	Central					9
10	Carpenters and helpers					10
11	Cranemen					11
12	Drill foreman					12
13	Drillers					13
14	Drill helpers					14
15	Drill laborers					15
16	Drill mechanics					16
17	Dumpmen					17
18	Engineers and surveyors					18
19	Electricians					19
20	Engineers, locomotive					20
21	Engineers, steam shovel					21
22	Engineers, stationary					22
23	Firemen, locomotive					23
24	Firemen, steam shovel					24
25	Firemen, stationary					25
26	Foremen, steam shovel					26
27	Foremen, tracks					27
28	Foremen, surface					28
29	Laborers and bosses					29
30	Masons and plasterers					30
31	Master mechanics					31
32	Machinists and helpers					32
33	Machinemen and helpers					33
34	Miners					34
35	Oilman and helpers					35
36	Officemen					36
37	Pumpmen					37
38	Plumbers and pipe fitters					38
39	Painters					39
40	Pitmen					40
41	Repairmen					41
42	Samplers					42
43	Switchtenders					43
44	Stableman					44
45	Sandmen					45
46	Teamsters					46
47	Topmen					47
48	Trammers					48
49	Timber framers					49
50	Trackmen					50
51	Watchmen					51
52	Water and coal tenders					52
53	Yardmasters					53
54						54
55						55
	Total					
1						1
2						2
3						3
4						4
5						5
	Total					
Ore						
Waste						

---

[a] Original form 20½ inches wide by 34 inches high. Original form has 20 instead of 4 distribution columns.

Another group of headings on these sheets indicate the following accounts: Water supply, Santa Rita boiler, compressor expense, heating, machine shop, blacksmith shop, carpenter shop, light service, engineering and surveying, assaying and sampling, office expense, pit drainage, stable expense, superintendent and general labor, and a few other accounts. Another group of accounts comes under the general term "Additions to plant," and the individual columns are headed "Crusher bins," "Hospital addition," etc. A fifth group is known as surface-development accounts. They include surface drainage, additions to dump lines, company roads, etc. The accounts-recoverable group comprises mess accounts, county roads built or repaired, etc.

Each column on each of these sheets (Form 22) represents a separate account. When these columns have been added, the total for each is divided between ore and waste according to the amount of time spent by each shovel in digging ore and the time spent in digging waste. These time figures are obtained from the reports of the shovel engineers, and are checked against foremen's reports, etc.

From the data recorded on the other forms the operating-cost sheets for the month (Form 23) are made out. One of these is used to show mining costs and another to show stripping costs. The expense apportioned to each is obtained from the total time spent by the shovels in digging waste and the total time spent by them in digging ore. The percentage of the total time spent in digging ore multiplied by the total cost for the month gives the cost of mining for the month. The remainder is the total cost of stripping. The recapitulation at the bottom gives a complete summary of the cost of production to date as well as costs for drilling, blasting, loading, and hauling. Thus, Form 23 provides a convenient and concise summary of all the costs.

FORM 23.—*Operating-cost sheet.*[a]

.... REPORT .... ORE. .... 191..

	Steam shovel No. 1.	Steam shovel No. 10.			Locomotive No. 1.	Locomotive No. 20.			Dump cars.	Air drilling.	Churn drilling.				Total cost.	Average per......
LABOR.																
Bankmen																
Boilermakers																
Blacksmiths																
Blasters and powdermen																
Brakemen																
Carpenters																
Car trimmers																
Car repairers																
Cranemen																
Drillers and helpers																
Dumpmen																
Electricians																
Engineering																
Engineers																
Firemen																
Floating gang																
Foremen																
Machinemen and helpers																
Machinists																
Officemen																
Oilmen																
Pipefitters																
Pitmen																
Pumpmen																
Sandmen																
Switchtenders																
Teamsters																
Track gang																
Watchmen																
Water and coal tenders																
Yardmaster																
Assaying and sampling																
Stable expense																
Superintendent and general labor																

Compressor expense																
Water supply																
Surface expense																
S. R. pumping and repairs																
Total labor																
Cost per																
SUPPLIES.																
Caps, fuse, and explosives																
Drill steel																
Electrical supplies																
Fuel																
Gasoline																
Miscellaneous																
Oil, waste, and packing																
Pipe and fittings																
Repairs and renewals																
Tools																
Teaming																
Office expense																
Engineering expense																
Assaying and sampling																
Stable expense																
Superintendent and general labor																
Compressor expense																
Water supply																
Surface expense																
S. R. pumping and repairs																
Total supplies																
Cost per																
Total labor and supplies																
Cost per																
No. cars loaded																
No. .... loaded																

[a] Original sheet, 17 by 31¼ inches.

Form 23.—*Operating-cost sheet*—Continued.

RECAPITULATION.

	Previously reported.				Cost this month.				Total to date.				Cost per............					Remarks.
	Amount.	No. of cars.	No. of	Cost per	Amount.	No. of cars.	No. of	Cost per	Amount.	No. of cars.	No. of	Cost per	Drilling.	Blasting.	Loading.	Hauling.	Total.	
Labor																		
Supplies																		
Total																		
Administrative expense																		
Grand total																		

WAREHOUSE METHODS.

The importance of the warehouse or storehouse division will be grasped from the fact that it always carries on hand and accounts for about a quarter of a million dollars' worth of stock of most varied character, say from turpentine to teeth for steam shovels.

All invoices received during the month are recorded in triplicate, Form 24, a "statement of invoices." The original of these is sent to the cashier's office, a duplicate goes to the supply agent, and the triplicate is retained in the warehouse. The invoices are then entered on the "warehouse-record" sheets (Form 25). These give a record of the freight charges, the material actually received, and the material invoiced. From this form the material is entered on large "commodity distribution" sheets, where the data covering different commodities are segregated in different columns, so that the amount of each received is easily determined. The commodities are then entered in the "price-and-invoice" book (Form 26), each page being given over to a different article, such as cylinder oil or steel rails. The price and balance on hand is kept posted for each month, and in addition stock is taken in December of each year and checked against this book.

Data covering the supplies issued are carefully recorded in several ways. Form 27 is an order on the storekeeper for material. The data on each of these forms received are entered in the "supplies issued record" book (Form 28), which shows the kind, quantity, and cost of supplies issued for every day of each month. At the end of the month these daily records are summed up on a monthly record known as the "Santa Rita supply distribution" sheet (Form 29). This summarizes the supplies charged during the month and furnishes part of the data for the work of the cost accountant in the main office.

The warehouse also keeps a record of the powder supply. Every week the powder supply is balanced in pounds of weight and every month in money. Form 30 is used for this record.

Many other labor-saving forms are used, but as they pertain more to general warehouse accounting than to mine accounting, they are not introduced here.

FORM 24.—*Form used in making statement of invoices.*[a]

STATEMENT OF INVOICES.

SANTA RITA MINE, ..., 191 . .

Date.	Favor of—	Amount of invoice.	Over-charges or deductions.	Amount taken into account.	Local freight bill No.	Amount of freight.
	Total					

[a] Original form 8½ inches wide by 14 inches long.

FORM 25.—*Warehouse-record sheet.*[a]

WAREHOUSE RECORD.

SANTA RITA MINE, ... month, 191 . .

Freight bill.							Material received.			Invoice.		
Date.	Local freight bill No.	Car initial and No.	Weight.	Rate.	Amount.	Date passed, month entered.	Date received.	Quantity and weight.	Consignor, shipping point, and description.	Net amount.	Date of invoice.	Date passed, month entered.

[a] Original sheet 13¾ inches wide by 16 inches long.

FORM 26.—*Specimen sheet from price-and-invoice book.*[a]

Nuts, bolts, and washers.

	Jan.	Feb.	Mar.	Oct.	Nov.	Dec.	Bal. for'd.
Avg. price							
New price							
Bal. on hand							
Rec'd							
Total							
Used							
Order No							

FORM 27.—*Form used in order on storekeeper for material.*[b]

REQUISITION FOR SUPPLIES.

No.......[of foreman].

SANTA RITA, N. MEX.,......................191..

To STOREKEEPER:

No.	Material.	For—

Storekeeper Req. No. Approved.

....

Signed.

FORM 28.—*Form used in recording daily issues of supplies.*[c]

SUPPLIES ISSUED RECORD.

Month of......................191.. ....Account.

1	2	3	4	5	6	29	30	31	Articles.	Total.	Price.	Amount.	Total.
											$ ¢	$ ¢	$ ¢

---

[a] Original sheet 15 inches wide by 12½ inches high; sheet printed on both sides. Eight spaces instead of one space appear for "Order No." entries; also columns for each month of the year.

[b] Original form 8½ inches wide by 10½ inches high, printed on white paper. A duplicate set of forms is printed on yellow paper, a yellow duplicate of each order being retained by foreman making requisition.

[c] Original form, 17 inches wide by 11 inches high. Margin and punch holes at left provided for binding; also columns for each day of the month.

FORM 29.—*Form used in summarizing daily records from Form 28.*[a]

SANTA RITA SUPPLY DISTRIBUTION.

Sheet No.......... ....191..

		On hand. (b)	Received. (c)	Used. (c)	On hand. (d)	
1	Air-drill repairs					1
2	Air-brake repairs					2
3	Babbitt and metals					3
4	Belting					4
5	Boiler flues					5
6	Boiler compound					6
7	Blacksmith coal					7
8	Burlap and canvas					8
9	Candles					9
10	Cement, brick, and lime					10
11	Castings					11
12	Caps					12
13	Coke					13
14	Drill repairs					14
15	Dump-car repairs					15
16	Electrical fixtures and supplies					16
17	Fuse					17
18	Fire brick and clay					18
19	Fuel					19
20	Furniture and fixtures					20
21	Grease and lubricants					21
22	Hay, grain, and feed					22
23	Hose and fittings					23
24	Hardware					24
25	Iron and steel					25
26	Illuminating oils					26
27	Laboratory supplies					27
28	Lumber					28
29	Locomotive repairs					29
30	Labor					30
31	Machinery repairs					31
32	Metal lath					32
33	Nuts, bolts, and washers					33
34	Nails					34
35	Packing					35
36	Pipe and fittings					36
37	Pulleys and shafting					37
38	Powder					38
39	Paint, tar, and glass					39
40	Pump repairs					40
41	Rail and fittings					41
42	Rope and cable					42
43	Screens					43
44	Steam-shovel repairs					44
45	Stationery					45
46	Structural steel					46
47	Sand					47
48	Switching					48
49	Ties					49
50	Teaming					50
51	Timber and lagging					51
52	Tools					52
53	Waste					53
54						54
55						55
	Total					
Santa Rita store supplies						
Electric power from Hurley						
1						1
2						2
3						3
4						4
5						5
	Total					
Ore						
Waste						

---

[a] Original form, 24 by 24 inches. Original has 20 instead of 2 blank columns for headings showing distribution of supplies.

[b] At first of month.

[c] During month.

[d] At first of succeeding month.

FORM 30.—*Form for keeping record of powder supply.*[a]

POWDER SUPPLY.

....................191..

Powder.	1st to 7th.			8th to 14th.			15th to 21st.			22nd to			1st of			
Grade and size.	On hand 1st.	Rcd. during period.	Used during period.	On hand 8th.	Rcd. during period.	Used during period.	On hand 15th.	Rcd. during period.	Used during period.	On hand 22nd.	Rcd. during period.	Used during period.	On hand 1st.	Used during month.	Orders not rcd.	
															Order No.	Amount.
Bulk and Total black Total stick Grand total																

---

[a] Original form, 16 inches wide by 8½ inches high.

PUBLICATIONS ON MINE ACCIDENTS AND METHODS OF METAL MINING.

Limited editions of the following Bureau of Mines publications are temporarily available for free distribution. Requests for all publications can not be granted, and applicants should select only those publications that are of special interest to them. All requests for publications should be addressed to the Director, Bureau of Mines, Washington, D. C.

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BULLETIN 62. National mine-rescue and first-aid conference, Pittsburgh, Pa., September 23–26, 1912, by H. M. Wilson. 1913. 74 pp.

BULLETIN 75. Rules and regulations for metal mines, by W. R. Ingalls, James Douglas, J. R. Finlay, J. Parke Channing, and John Hays Hammond. 1915. 296 pp., 1 fig.

BULLETIN 80. A primer on explosives for metal miners and quarrymen, by C. E. Munroe and Clarence Hall. 1915. 125 pp., 15 pls., 17 figs.

BULLETIN 101. Abstracts of current decisions on mines and mining, October, 1914, to April, 1915, by J. W. Thompson. 1915. 138 pp.

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TECHNICAL PAPER 6. The rate of burning of fuse as influenced by temperature and pressure, by W. O. Snelling and W. C. Cope. 1912. 28 pp.

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FRANKLIN K. LANE, SECRETARY
BUREAU OF MINES
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MELTING ALUMINUM CHIPS

BY

H. W. GILLETT AND G. M. JAMES

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MELTING ALUMINUM CHIPS.

By H. W. Gillett and G. M. James.

INTRODUCTION.

In its work on mineral wastes the Bureau of Mines is studying losses in the melting of nonferrous metals and alloys. The greatest of these losses is that of zinc through volatilization in brass melting, but another and considerable loss is that of aluminum and its alloys in the melting of finely divided material.

In order to get some idea of the magnitude of the loss in melting aluminum scrap and to find out what methods of melting were used information was gathered in regard to present practice; also experiments were conducted to obtain some data on the relative merits of different methods. As the experiments could be made on a laboratory scale only, they were not expected to solve the problem entirely, the object in making them being rather—by indicating which methods were of greater promise—to narrow the field over which large-scale tests and comparisons need be made by those commercially nterested in the problem of melting scrap aluminum.

NATURE OF SCRAP ALUMINUM.

Scrap aluminum may be produced in the fabrication of sheet aluminum into various objects, such as cooking utensils, camera frames, and various ornamental goods, yielding sheet clippings or punchings, in the machining of aluminum castings for automobiles, motor boats, cooking utensils, vacuum cleaners, meter cases, and the multitudinous other articles in which cast aluminum is used, yielding borings or chips, or in the foundry, in the chipping and grinding of rough castings, and in the recovery of buttons of metal from foundry sweepings, or from the dross skimmed from the melting pots.

CLIPPINGS AND PUNCHINGS.

Sheet clippings and punchings do not, as a rule, show a very great melting loss, for they are normally kept clean and are usually not excessively small nor thin, and may without the use of very great pressure be "cabbaged" or pressed into brick-like bundles, which are considered almost as valuable for foundry use as ingot metal, particularly as it is necessary, in order to obtain good rolling qualities, to use purer metal for sheet than is sold for casting purposes. "Hard-sheet" aluminum, containing upwards of 1 per cent of manganese, must of course be used with due consideration of the content of the alloyed metal.

SCRAP WIRE AND CABLE.

Scrap wire and cable is a still purer aluminum than unalloyed sheet aluminum, as the very purest metal attainable is required for high electrical conductivity. Bare wire and cable is a desirable form of scrap, but insulated wire would be hard to handle in remelting. On account of the growing use of aluminum wire of small diameter, that is electrolytically insulated with a film of aluminum oxide,[a] this wire will at some future time form a variety of scrap that will be more or less difficult to remelt.

CASTINGS, CHIPS, AND BORINGS.

The great bulk of the scrap aluminum met with at present consists of old castings and of chips and borings obtained in machining castings. Melting down old castings presents no difficulty, as the loss is scarcely greater than with ingot metal. The chips and borings, however, give much trouble, and form the main problem in regard to metal losses in remelting scrap aluminum.

As at least 95 per cent of all cast-aluminum alloys is used for automobile engine beds or crank cases, gear cases, transmission cases, footboards, bodies, steering-wheel spiders, intake and outlet manifolds, hub caps, and miscellaneous trimmings and fittings, chips from a motor-car manufacturing plant may be regarded as typical.

The drill presses and milling machines used on aluminum castings run either with a light cutting oil, kerosene being often used, or with cutting lubricants of the general type of emulsions of oil in a soap solution. The emulsions seem to be coming into increasing use.

If the chips stand in the air while wet with such aqueous solutions, that is, if the solution is not drained off at once, they become superficially oxidized and caked together.

The chips become covered with whatever lubricant is used, soaking it up like a sponge. If they fall on the floor and are swept up without due care to keep them separate from the floor dust, the dust sticks to the oily or moist borings.

If the machining of iron, steel, brass, or white metal is done near by, chips of those metals are likely to become mixed with the aluminum chips. It is the exception and not the rule to find aluminum chips that are not contaminated with a few per cent of iron chips. The iron necessitates passing the chips over a magnetic separator. On account of the more curly form of chips from aluminum than from most commercial brasses and bronzes, it is harder to effect a complete separation of the iron, particularly from damp or oily chips.

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[a] Skinner, C. E., and Chubb, L. W., The electrolytic insulation of aluminum wire: Trans. Am. Electrochem. Soc., vol. 26, 1914, p. 137.

The chips may go to the aluminum foundry of the automobile plant, if it makes its own aluminum castings, or may be sent to various refiners, who either buy the chips outright or refine them into ingots, make a smelting charge, and return the ingots obtained, or the chips may be sold in small lots to some small dealer in scrap metal, who adds them to various other chips until he has enough to melt down himself or sell to a larger operator.

Some of the larger operators state that it is apparently difficult for some of the small junk dealers to resist the temptation to add road dust, foundry sweepings, or other fine dirt to the borings, as 10 per cent or more will readily stick to oily borings and be scarcely detectable even on close inspection.

The size and thickness of the chips depends on the depth of the roughing and finishing cuts. The thickness commonly runs between 0.005 and 0.02 inch.

RECOVERY ON SCRAP ALUMINUM.

Foundry wastes, such as spillings recovered in floor sweepings, small fins that work their way into core prints, chippings from the trimming room, and dust from the emery grinders, are all badly contaminated with dirt. The larger pieces are picked out by hand and the rest concentrated by tumbling dry in a stream of air, or in a wet tumbling barrel. The scum of oxide, dirt, and metal that is skimmed from the melting pots is generally remelted in the foundry, usually with the use of sal ammoniac or zinc chloride, and the dross from this remelting is thrown away, sold to a refiner, or dropped into water as soon as skimmed off, and then subjected to wet tumbling.

In melting fine material, such as borings or chips, fine shot from foundry wastes, and dust from grinding wheels, all heavy scrap being excluded, the recovery as ingot metal runs from 40 to 90 per cent of the metal content, the proportion recovered varying with the size and cleanness of the material and with the facilities, experience, and method of the melter.

It is very doubtful if any but the largest and most experienced refiners average more than 65 per cent recovery from the ordinary dirt-laden borings of commerce. One firm organized solely to refine borings to ingot suspended operations after a few months, and another firm has run its aluminum-refining department only intermittently, finding that when aluminum was scarce it was better to stop refining borings because their price rose above that at which a profit could be made with the percentage of recovery obtained. At such times the borings go to small refiners, or to foundries that refine the borings separately or add them to the regular melts. In many cases no accurate records of costs are kept by such plants, and the operators may be refining at a considerable loss without knowing it.

A prominent aluminum founder states that with pure aluminum selling at 25 cents per pound and copper at 15 cents, the most common casting alloy, "No. 12," containing 8 per cent copper, would sell in new ingot for about 24½ cents, whereas good No. 12 borings would command about 12½ cents and the ingot made from the borings would sell at about 22 cents. If the cost of refining, including the cost of fuel, labor, overhead charges, etc., is arbitrarily assumed to be 1 cent per pound of ingot produced, it will be seen that with a 50 per cent recovery on the gross weight of borings bought, the cost of producing a pound of ingot would be 25 cents for metal and 1 cent for refining charges, or 26 cents, which, with metal selling at 22 cents, would result in a loss of 4 cents per pound of ingot, whereas with a 70 per cent recovery the cost would be about 20 cents, netting a profit of 2 cents per pound. With a 60 per cent recovery there would be a slight loss, whereas with an 80 per cent recovery the profit would be over 5 cents per pound. It is assumed that the same price, 12½ cents, is paid for borings from which only 50 per cent of metal is recovered as for those yielding 80 per cent.

If it be assumed that the average recovery from chips and foundry waste is 65 to 70 per cent, on the metallic basis, and if, as is shown later, borings kept perfectly clean and properly melted will give 90 to 95 per cent recovery, then 20 to 30 per cent of the aluminum that is machined off castings is unnecessarily lost.

The probable amount of aluminum castings used in motor-car construction from July 1, 1914, to July 1, 1915, has been estimated by the general manager of a firm of aluminum founders, whose production alone is over half that amount, to be between 20,000,000 and 25,000,000 pounds. Addition of heavy export orders to the increasing domestic demand will undoubtedly cause the amount used from July 1, 1915, to July 1, 1916, to exceed 25,000,000 pounds.

The percentage machined off from a rough aluminum casting in the finishing process seems, from data obtained by the writer, to average about 15 per cent, a figure which is much higher than was estimated offhand by several people connected with the automobile and aluminum casting industries, whose guesses averaged 3 to 4 per cent. The writer's result was reached by weighing rough and finished castings at an automobile plant making a car in the $700 to $800 class, and by weighing rough and finished castings made for various other motor-car manufacturers at an aluminum foundry, with the following results:

Proportion of metal machined off in finishing alumnium castings for motor cars.

Casting.	Weight of rough casting.	Weight of finished casting.	Percentage of metal machined off.
	Pounds.	*Pounds.*	
Gear and transmission case	28.50	24.50	14
Small double-flanged exhaust elbows	.42	.35	15
Intake pipe for 6-cylinder motor	5.00	4.80	4
Gear case for small motor	8.90	7.00	21
Gear case for very small motor	1.58	1.50	5
Crank case for 8-cylinder motor	94.00	80.00	14.5

As crank cases and transmission cases make up the bulk of the weight of aluminum castings in a car, the values obtained from them will have more effect on the total than those obtained from the smaller castings.

The results of this tabulation, which indicate that 15 per cent was machined off, were so at variance with the estimates cited that the matter was taken up with the makers of a car in the $2,000 class, who state that on that car there are used 47 aluminum parts which are machined, the total weight of the rough castings used per car being 166.19 pounds. The loss per car in chips is 25.15 pounds, or 15.1 per cent. The weights were taken on the average of a number of like parts, in some cases as many as 50 being weighed. On the basis of 15 per cent of metal machined off, the yearly production of chips from aluminum castings in the United States will then run between 3,000,000 and 3,750,000 pounds. If 20 to 30 per cent of this amount is unnecessarily lost, then 600,000 to 1,125,000 pounds of aluminum alloys worth 16 to 25 cents per pound, based on the average market quotations for the last few years, is lost, or, as these figures take no account of the loss in running down foundry wastes, it may be estimated that in all metal worth approximately $200,000 at average prices for aluminum, is lost each year in the United States through the low recovery in melting down aluminum borings and foundry waste. At the abnormal price of aluminum in 1916 this figure would be doubled or trebled.

This estimate is not too high, as the United States Geological Survey states[a] that in 1913 2,198 tons of secondary aluminum and 2,456 tons of secondary aluminum alloys (90 per cent of the latter being "No. 12" alloy, containing 92 per cent aluminum and 8 per cent copper) were recovered, mostly from aluminum clippings and borings, the whole having a value of $2,199,480. The total consumption of aluminum in the United States in 1913 was 36,190 short tons.[b]

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[a] Phalen, W. C., Bauxite and aluminum: Mineral Resources of the United States for 1913, U. S. Geol. Survey, 1914, p. 16.

[b] Phalen, W. C., Loc. cit.

If the pure aluminum, mostly from clippings, which give low loss on melting, be entirely disregarded and the alloys only be considered, and if it be further assumed that only two-thirds of the quantity of alloys came from borings, there would be about 3,250,000 pounds of secondary aluminum alloys recovered. If this figure represents a 70 per cent recovery of the original metal content in the borings, which is as high as is probable, and if a 90 per cent recovery is possible then two-sevenths of 3,250,000 pounds, or over 1,000,000 pounds, was lost in melting. The average value of the secondary pure aluminum and alloys was about 23½ cents per pound in 1913, according to the figures of the Geological Survey. If the alloy is assumed to be worth 20 cents, the value of the lost metal figures to about $200,000 for 1913.

The 1914 figures[a] show 2,791 tons of secondary aluminum and 1,731 tons of recovered aluminum contained in alloys, having a total value of $1,673,140, or an average of 18½ cents per pound. It is stated that the greater part of the secondary aluminum was recovered from clippings and borings. Pure aluminum borings are not common, and as refineries commonly term borings from aluminum alloys "aluminum borings," their reports may not have made a clear distinction between pure and alloyed metal.

Although figures for 1914 showed a decrease in amount and value of recovered aluminum alloys as compared with 1913, the high average price of aluminum in 1915 will bring the values of the losses for 1915 to about $300,000, as in the fall of 1915 the price of aluminum rose to about three times the prices quoted in 1914 and in the spring of 1915.

There are, of course, other possible uses for aluminum alloy chips than as a source for the recovery of ingot metal. Their use for the manufacture of aluminum paint at once suggests itself, but it is doubtful if all alloys of aluminum are malleable enough to produce the very thin flat flakes required in aluminum paint. Pure aluminum scrap could doubtless be so used. One automobile firm, during the period of unusually high prices for aluminum in 1915 and 1916, was forced to find a substitute for the aluminum paint previously used on its motors and found one in finely crushed ferrosilicon. This firm also reported that it could, in this period, obtain a higher price for its aluminum chips from a chemical company than it could from the metal refiners. The chips are evidently used in the preparation of anhydrous aluminum chloride, to be used as a condensing agent in the manufacture of dyes.

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[a] Phalen, W. C., The production of bauxite and aluminum: Mineral Resources of the United States for 1914, U. S. Geol. Survey, 1915, pp. 183-200.

METHODS OF MELTING ADVOCATED IN THE LITERATURE.

Various methods of melting down chips are suggested in the periodical and patent literature. One of the earliest comments[a] on the subject is as follows:

ADDING SCRAP TO MOLTEN METAL.

We recently had an inquiry for a method of recovering the aluminum from the skimmings taken from the casting ladle. The company making the inquiry is in the aluminum-casting business, the parts being mostly for automobile work. One essential was that the recovered metal must be of such degree of purity that it could be used over again for casting. As the quantity of skimmings is large, the waste at present going on is considerable.

There is no way of accomplishing this in a commercial manner except by putting the skimmings into the reduction pots, and even that is objectionable with a great many kinds of alloy skimmings, particularly those containing zinc. It is extremely difficult to get finely divided particles of aluminum to agglomerate, even when fused, owing to the difficulty of breaking the skin which covers each particle. The only method by which it was ever done is that recited by De Ville—namely, melting finely divided metal by using a double chloride of sodium and aluminum, which acts as a flux and permits some of the finely divided aluminum to be made into a button. This operation is, however, so difficult and so unusual that the expense of it is considerably more than the value of the metal recovered.

Sperry[b] states "The best way to melt aluminum scrap, such as sheet or chips, is to have a pot of molten aluminum at low red heat and then to add the scrap to it, pushing it down at once under the surface, so that it is not exposed to the air at all. In this manner there is no oxidation of the aluminum and it can be melted with the minimum waste. The scrap will dissolve in the molten aluminum, and this is an ideal condition." He also states[c] that the chips should be added in small quantities at a time to already molten metal with immediate stirring, and the pot fluxed at the end of the melt with a little zinc chloride.

Vickers[d] suggests practically the same method. Still another description[e] of this general method follows:

Question.—We have several barrels of aluminum turnings and borings and would like to know the best method of converting this material into pig form. We understand that under certain conditions of melting a considerable loss of aluminum results.

Answer.—The melting of finely divided metals entails considerable work, and furthermore, the material must be handled in a manner which will prevent undue oxidation. The best way to accomplish this is to dissolve the finer metal in a bath of molten metal. A large crucible should be used and the molten metal should be obtained by melting ingots or other bulky stock. In the case of aluminum, the bath should be raised to a temperature not above red heat. Charge as many borings at a time as the bath will dissolve, but do not leave any of the borings on top of the bath in a solid state. These should be stirred into the molten metal. Inasmuch as the bath increases with each charge of borings, it will dissolve more each time that additions are made. As the borings will cool the bath, the furnace should be closed from time to time, the metal again heated to redness, when more borings can be added, and this process can be continued alternately until the pot is full. At this stage the metal is cast into ingots, but sufficient should be left in the pot to form another bath. If the mush-like dross gathers on the surface of the metal, add a small piece of fused zinc chloride and stir it on the surface. This will liberate the metal from the dross, which will form a cover for the aluminum. The metal should be skimmed before casting into ingots. Incidentally, this dross is of no value.

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[a] Editorial, Recovery of aluminum from skimmings: Metal Ind., vol. 5, 1907, p. 100.

[b] Sperry, E. S., Questions and answers: Brass World, vol. 9, 1913, p. 296.

[c] Sperry, E. S., Remelting of aluminum chips or borings; Brass World, vol. 6, 1910, p. 278; Jour. Inst. Metals, vol. 5, 1911, p. 342.

[d] Vickers, C., How to melt aluminum turnings or borings: Foundry, vol. 41, 1913, pp. 119, 420.

[e] Editorial, Recovery of aluminum turnings and borings, problems of the brass founder: Foundry, vol. 43, May, 1915, p. 195.

In doing this work the borings should be passed through a magnetic separator to remove the iron, and under no conditions should the bath of molten metal be heated above redness at any time, as otherwise the aluminum will attack the silica in the crucible and the metal will contain black specks.

A similar method in which turnings are to be charged into a "heel" of molten metal, but a reverberatory furnace used, not a crucible, has been advocated,[a] though no mention is made of stirring or the use of any flux.

Echevarri[b] says, "Scrap can be remelted by submerging in the molten metal, the loss being only 2 to 3 per cent." This statement evidently refers to heavy scrap.

Coulson[c] has recently described a puddling method, commercially used for the recovery of turnings from an aluminum alloy with 5 to 10 per cent of magnesium, as well as considerable experimental work on the problem. Previous to the experimental work, cryolite had been used as a flux, the recovery with clean turnings varying from 60 to 90 per cent on clean material. In the experiments, turnings were melted in air-tight, electrically heated furnaces, in which the air could be displaced by reducing gases, such as hydrogen. Even in such a nonoxidizing atmosphere 10 per cent was lost, and nitrides were still formed, indicating that air was occluded in the borings. Then the turnings were boiled in a 4 per cent salt solution to displace occluded air and wash off dirt. These treated borings, melted under hydrogen, still gave 8 per cent loss. Melting under hydrogen was considered impractical for commercial work, so melting in open crucibles was tried. The borings were washed in benzine and then boiled in a salt solution. The damp borings were fed into a hot crucible containing a small "heel" of molten metal, and each addition thoroughly puddled until the mass became uniformly viscous. After all the borings were in, the charge was allowed to stand until the dross had risen, when the clean metal was poured from beneath the dross, or tapped off from a hole in the bottom of the crucible.

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[a] F. H. H., Refining scrap: Foundry Trade Jour., vol. 14, 1914, p. 29.

[b] Echevarri, J. T. W., Aluminum and some of its uses: Jour. Inst. Metals, vol. 1, 1909, p. 128.

[c] Coulson, J., Reclamation of magnalium from turnings: Trans. Am. Inst. Metals, vol. 9, 1915.

Calcium chloride was then stirred into the dross. If the temperature rose through thermit action, powdered cryolite was stirred in. The metal separated from the dross was then poured or tapped. The loss by this method averaged 6 per cent for small melts, and with 200-pound melts of clean material the loss has been as low as 1 per cent.

It is stated that the addition of 1 per cent of calcium or 0.5 per cent of calcium aluminum silicide restores the reclaimed metal to its original physical state as shown by tensile tests.

Such additions should be made with caution, since for some purposes the presence of alkali or alkaline earth metals is detrimental, and in the production of aluminum every effort is made to keep them out, as they are considered highly undesirable impurities. Le Chatelier [a] has shown that calcium aluminum alloys are liable to disintegrate, apparently spontaneously.

Puddling methods were used by De Ville on a small scale in 1859, and his directions, as quoted by Richards,[b] quite closely describe the puddling method in use to-day. These directions are as follows:

> When it is desired to melt pieces together, they can be united by agitating the crucible or compressing the mass with a well-cleaned cylindrical bar of iron. Clippings, filings, etc., are melted thus: Heat the divided metal to as low a heat as possible—just sufficient to melt it. The oil and organic matter will burn, leaving a cinder, which hinders the reunion of the metal if the mass is not pressed firmly with the iron bar.

USE OF FLUXES IN MELTING ALUMINUM SCRAP.

Zinc chloride ($ZnCl_2$) is often used as a flux in running down chips and skimmings, as well as in ordinary melting, it being claimed that the zinc chloride aids the separation of the metal from the dross so that instead of a pasty mass of metal plus dross only a dry, crumbly dross is taken off.[c] The value or uselessness of zinc chloride as a flux for ordinary melting is a matter of contention among aluminum founders. Lane [d] found that 0.015 per cent of zinc was taken up by an aluminum alloy on one melting and 0.085 per cent after the eighth remelt, zinc-chloride flux being used each time. He suggests that some of the zinc chloride may be decomposed by the heat and part of the zinc may alloy with the aluminum, the nascent chlorine being the active agent of the flux.

Micks [e] states—

> The flux that has given the best results in aluminum is chloride of zinc. The zinc combines with the oxygen which is taken up from the aluminum oxide and forms zinc

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[a] Le Chatelier, H., Alterability of aluminum: Compt. rend., t. 152, 1911, p. 650; Chem. Abs., vol. 5, 1911. p. 1898.

[b] Richards, J. W., Aluminum, its properties, metallurgy, and alloys, 1890 pp. 202, 247.

[c] Anon., Use of zinc chloride in melting aluminum: Jour. Inst. Metals, vol. 2, 1909, p. 320.

[d] Lane, H. M., Use of magnesium in deoxidizing aluminum alloys: Trans. Am. Brass Founders' Assn., vol. 4, 1910, p. 101; Castings, vol. 7. 1911, p. 164.

[e] Micks, R., Furnace practice in the brass foundry: Brass World, vol. 11, 1915, p. 150.

oxide, which is then skimmed off together with the aluminum chloride that is also formed in the reacton when the flux is added. The best time to add this flux is after the melting is completed; the surface of the molten metal is then covered with a mass of dross, but when the chloride of zinc is dropped on it in small pieces it will become clear as soon as enough of the flux has been added.

Lane considers that magnesium is a deoxidizer of aluminum. However, Matignon[a] reports the reduction of magnesium oxide by aluminum. It is also stated[b] that although from thermochemical data both magnesium and calcium should reduce aluminum oxide, magnesium does not act as strongly in a thermit mixture as aluminum, and that it probably is not a deoxidizer for aluminum. It was thought that calcium was reduced by aluminum when calcium chloride was used as a flux, and the statement was made that the only value of calcium chloride or zinc chloride as a flux is in disentangling the aluminum from the dross.

Another flux recommended[c] is anhydrous aluminum chloride ($AlCl_3$), the claim being made that by the use of a small amount of aluminum chloride a recovery of 50 to 70 per cent could be obtained from chips, which without it yielded only 30 per cent.

Another flux recommended consists of sodium carbonate 10 parts, potassium carbonate 2 parts, cryolite 2 parts, and borax 1 part, to be used in the proportion of 1½ pounds per 100 pounds of chips.[d]

Borax is suggested by Mellen[e] as a flux to dissolve oxides of aluminum.

However, Richards[f] states that aluminum melted under borax is rapidly dissolved, an aluminum borate being formed.

Weber[g] suggests the use of fluorides of zinc, copper, or nickel, mixed with alkali fluorides. The aluminum reduces the fluoride of the heavy metal used, the zinc, copper, or nickel alloying with the aluminum and forming an equivalent amount of aluminum fluoride. The mixture of aluminum fluoride and alkali fluorides then acts as a slag to dissolve the aluminum oxide. Weber also suggests a mixture of zinc chloride and sodium fluoride.

Jones[h] suggests a flux of equal parts of lithium chloride, potassium chloride, and sodium fluoride for use in welding aluminum.

Schoop[i] emphasizes the difficulty of breaking through the thin surface film of oxide in welding aluminum so as to make the fluid metal, beneath the films of the two pieces to be welded, unite, and states that he has tried borax, glass, and other substances to exclude the air, but found that a flux which would dissolve the oxide was needed. He then tried potassium pyrosulphate and many other substances as fluxes to dissolve the oxide, but found a satisfactory solution of the problem only in the use of alkali chlorides. In his patents Schoop[a] mentions a mixture consisting of 60 parts potassium chloride, 20 parts lithium chloride, 12 parts sodium chloride, 8 parts potassium sulphate. In a later patent[b] he substitutes cryolite for the potassium sulphate, and in another,[c] he says, "Experiments have shown that the said defects may be completely avoided by adding fluorides—for example, fluorides of calcium, potassium, or boron to the mixture of chlorides of alkali metals. By means of the improved flux it is possible also to melt down or fuse together in crucibles small and large scraps of aluminum in the shape of castings, sheets, wires, or other objects."

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[a] Matignon, C., Reduction of magnesia by aluminum: Compt. rend., t. 156, 1913, p. 1157; Jour. Soc. Chem. Ind., vol. 32, 1913, p. 491.

[b] Anon., Oxidation of aluminum: Foundry, vol. 37, 1910, p. 225.

[c] Anon., The fusion of aluminum chips: Jour. Mines Met., t 1, 1912, p. 7; Chem. Abs., vol. 6, 1912, p. 2223; Brass World, vol. 9, 1913, p. 365.

[d] Anon., Melting aluminum chips: Castings, vol. 38, 1910, p. 78.

[e] Mellen, J. G., and Mellen, W. F., U. S. Patent 982218, Jan. 17, 1911.

[f] Richards, J. W., Aluminum, its properties, metallurgy, and alloys, 1890, p. 81.

[g] Weber, H., Process for melting scrap aluminum or alloys high in the metal: German Patent 242347, Dec. 30, 1910; Jour. Soc. Chem. Ind., vol. 31, 1912, p. 237; Chem. Abs., vol. 6, 1912, p. 2063.

[h] Jones, J. L., Shop problems—fluxing: Metal Ind., vol. 11, 1913, p. 528.

[i] Schoop, M. V., Autogenous welding of aluminum: Electrochem. and Met. Ind., vol. 7, 1909, p. 151.

Morrison[d] speaks well of such a flux for welding, as does Seligman.[e] Other welding fluxes have been suggested, Thaulow[f] recommending 96 parts of borax and 4 parts of sodium bisulphate.

Pannell[g] gives the composition of various fluxes for use in welding, as follows:

Composition of various fluxes for use in welding.

Flux No.	Constituent.							
	Sodium chloride.	Potassium chloride.	Lithium chloride.	Potassium fluoride.	Sodium fluoride.	Sodium bisulphate.	Potassium bisulphate.	Cryolite.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
1	30.0	45.0	15.0	7.0		3.0		
2		33.0	33.0		33.0			
3	12.5	62.5	20.8				4.0	
4	16.0	79.0					5.0	
5	17.0	83.0						
6	6.5	56.0	23.5			4.0		10.0

It is seen that these are all mixtures of alkali chloride high in the low-melting potassium and lithium chlorides, with or without the addition of fluorides or bisulphates.

Other processes are based on the theory that as aluminum is readily oxidizable, the melting should take place in the absence of air. Mellen[h] patented a vacuum furnace in which it is claimed that aluminum clippings, turnings, and filings ordinarily melted with a loss of 10 to 40 per cent may be melted with a loss of only 0.5 to 5 per cent.

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[a] Schoop, M. V., U. S. Patent 922523, May 25, 1909; English Patent 24096, Oct. 31, 1907; French Patent 374089, Jan. 30, 1907.

[b] Schoop, M. V., U. S. Patent 943164, Dec. 14, 1909.

[c] Schoop, M. V., English Patent 24283, Nov. 2, 1907.

[d] Morrison, W. M., In discussion: Jour. Inst. Met., (British) vol. 1, 1909, p. 150.

[e] Seligman, R., The welding of aluminum; Jour. Inst. Met. (British), vol. 2, 1909, p. 281.

[f] Thaulow, E., U. S. Patent 1139923, Mar. 4, 1914; Brass World, vol. 11, 1915, p. 181.

[g] Pannell, E. V., Recent developments in aluminum; some notes on autogenous welding: Trans. Am. Inst. Metals, vol. 9, 1915. Advance copy, p. 17.

[h] Mellen, G., U. S. Patent 1120732, Dec. 15, 1914.

One inventor, according to parties to whom the invention was offered for $100,000, had a plan to melt aluminum borings by melting in retorts similar to those used in zinc smelting and passing in steam when it was desired to check the heating and stop oxidation by air. A recovery of 85 per cent was claimed. No further details are available.

Zavelberg[a] suggests for zinc smelting or the recovery of other readily oxidizable metals heating the furance, discontinuing the heating, charging and closing the furnace, and doing the melting by the stored heat. Practically the same method has been used in the melting of aluminum-magnesium alloys for casting purposes in a tilting open-flame oil furnace, though it does not seem to have been tried on borings.

Richards[b] says:

> Fused common salt is used as a flux for aluminum. Fluorspar makes a good flux for the metal, especially in connection with cryolite or common salt, and possesses the property of dissolving the aluminum oxide with which the metal may be contaminated and which, by incrusting small globules, hinders their reunion to a button.

Various fluxes have been used in melting aluminum without having been especially mentioned in connection with borings.

Guertler[c] mentions a mixture of sodium and potassium chlorides.

Zinc chloride, a mixture of sal-ammoniac (ammonium chloride) and common salt, as well as saltpeter (potassium nitrate) are all mentioned.[d]

Schultz[e] also reports favorably on the use of saltpeter in melting aluminum alloys to get sound castings. It seems remarkable that the addition of so strong an oxidizing agent to so readily oxidizable a metal should be beneficial, and the most plausible explanation is that the gas given off by the decomposition of the saltpeter stirs the metal and is carried off with the other and harmful gases previously dissolved in the metal. Saltpeter has not been advocated for use on chips. Sal ammoniac alone is also used.[f]

Gleason[g] melts aluminum under a flux composed of 3 parts fluorspar and 1 part anhydrous boric acid. Mellen[h] uses a flux consisting of salt and sulphur.

Desch[i] mentions borax, which melts at about 650° C., and anhydrous carnallite (equimolecular amounts of potassium and magnesium chlorides), which melts at about 450° C., as forming suitable protecting layers for low-melting alloys. The melting point of the eutectic mixture (2KCl.LiCl) of potassium and lithium chlorides is given [a] as 350° C. and that of the lowest melting mixture of NaCl and LiCl (27 per cent by molecular weight of NaCl, 73 per cent LiCl) as 550° C.

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[a] Zavelberg, A., U. S. Patent 1136304; German Patents 226257, Feb. 23, 1908; 276364, Aug. 6, 1913· Chem. Abs. vol. 9, 1915, p. 594.

[b] Richards, J. W., Aluminum its properties, metallurgy and alloys, 1890, p. 80.

[c] Guertler, W., Metallographie, Bd. 2, Heft 1, 1913, p. 39.

[d] Editorial, Flux for aluminum alloy—problems of the brass foundry: Foundry, vol. 41, 1913, p. 420.

[e] Schultz, F., In discussion: Trans. Am. Inst. Metals, vol. 8, 1914, p. 132.

[f] Carritto, J. P., Making McAdamite metal; Foundry, vol. 41, 1913, p. 421.

[g] Gleason, E. D., U. S. Patent 1076973, Oct. 28, 1913.

[h] Mellen, G., U. S. Patent 1092935, Apr. 14, 1914.

[i] Desch, C. H., Metallography, 1910, p. 106.

Burgess [b] gives the melting point of an equimolecular mixture of NaCl and KCl as about 650° C.

A flux recently put on the market at 25 cents per pound is said by its introducers to act as a molten cover to protect the metal from the air and also as a flux to dissolve impurities from the melt. The directions state that 1 pound of flux should be used to 250 pounds of metal and scattered over the cold metal as much as possible, so that the metal will be protected while being heated. After all the metal is melted it should be stirred with an iron rod, when the flux will come to the top, as it melts below the melting point of aluminum and rises as soon as the metal is melted. For running down scrap or skimmings, the flux is to be used in the same way, but more must be used than when clean metal is melted, the amount depending on the content of impurities.

Cryolite (sodium aluminum fluoride) is sometimes used as a flux in running down borings, as this is the main constituent of the electrolyte used in the reduction of aluminum, and aluminum oxide is quite soluble in it. The melting point of cryolite (about 980° C.) is high for the purpose, and various substances might be added to lower it.

In the direct testimony of A. H. Cowles before the Circuit Court of the Northern District of Ohio, May 6, 1899, C. M. Hall is quoted as stating that KCl and NaCl fluxes do not dissolve oxides and other dirt from aluminum, but fluorides do.[c]

Wright [d] states that the electrolyte used in the production of aluminum is composed of cryolite (AlF_3NaF), aluminum fluoride, and fluorspar in the proportions of about 16 per cent CaF_2, 28 per cent (AlF_3NaF), 56 per cent AlF_3, the AlF_3 being made by treating bauxite with HF. This is evidently taken from the United States patent 400667 of C. M. Hall, whose various patents Nos. 400665, 400666, 400667, and 400766 cover AlF_3 plus fluorides of Na, K, Li, and Ca.

Pascal [e] intimates that cryolite and fluorspar are both used in the electrolyte. He finds that the eutectic mixture of CaF_2 and Al_2O_3

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[a] Zemczuzny, S., and Rambach, F., Schmelzen des alkali Chloride: Zeit. anorg. chem., Bd. 65, 1910, p. 403.

[b] Burgess, G. K., The measurement of high temperatures, 1912, p. 451.

[c] See also Cowles, A. H., The solubility of alumina in a bath of fused fluorides: Met. and Chem. Eng., vol. 11, 1913, p. 177.

[d] Wright, J., Electric furnaces and their industrial applications, 1908, pp. 186-187.

[e] Pascal, P., Die Elektrometallurgie des Aluminum, Das ternäre System Tonerde-Fluorite-Kryolith: Ztschr. Electrochem., Bd. 19, 1913, p. 610. Pascal, P. and Jouniaux, M., Études physico chimiques sur l' électrometallurgie de l' aluminum: Rev. met., t. 11, 1914, p. 1069; Bull. soc. chim. France, t. 15, 1914, p. 312; Chem. Abs., vol. 7, 1913, p. 2904, vol. 8, 1914, pp. 2529, 3269.

contains 27 per cent Al_2O_3 and melts at 1,270° C, whereas Lorenz[a] finds that it contains 33 per cent Al_2O_3 and melts at 940° C. Hence fluorspar is a solvent for alumina.

Pascal finds a ternary eutectic for cryolite, fluorspar, and alumina at the proportions 59.3 per cent cryolite, 23 per cent fluorspar, and 17.7 per cent alumina—a mixture which melts at 868° C.

However, in the published accounts[b] of laboratory experiments for making aluminum, natural cryolite, without the use of CaF_2 or AlF_3, seems to be the electrolyte used.

Neumann and Olsen mention a mixture of 8 parts cryolite to 1 of NaCl, and also appear to have added some AlF_3 to the electrolyte in some tests. The addition of NaCl to fluorides in the manufacture of aluminum is also mentioned by Minet.[c] A mixture of 40 parts AlF_3 and 60 parts NaCl, another of 20 to 50 parts cryolite and 80 to 50 parts NaCl, and still another of 35 parts AlF, 10 parts NaF, and 55 parts NaCl are suggested.

Plato[d] states that a mixture of 60 parts KCl and 40 parts KF melts at about 610° C., and a mixture of about 86 parts $CaCl_2$ and 14 parts CaF_2 melts at about 650° C.

That fluorspar was a flux for Al_2O_3 was stated by Mierzinski[e] as far back as 1855, and fluorspar is known to be a good flux for silicates, such as the dirt in most dirty borings doubtless is.

Fedotieff and Iljinsky[f] find that the eutectic mixture for NaF and AlF_3 consists of 36.5 per cent NaF and 63.5 per cent AlF_3 by weight and melts at 685° C. For the manufacture of aluminum they recommend a mixture of the formula $5NaF.AlF_3$; this mixture has a lower specific gravity than the eutectic, but has to be run at 900° C. in order that the metal may separate well. The use of CaF_2 is not advised, as its specific gravity is even higher than that of aluminum. A mixture consisting of 85 parts $CaCl_2$ and 15 parts CaF_2 that melts at about 650° to 660° C. and has a specific gravity of 2.5, slightly higher than that of molten aluminum, is mentioned by several workers[g] on the electrolytic production of calcium.

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[a] Lorenz, R., Jabe, A., and Eitel, W., Beiträge zur Theorie der Aluminumdarstellung; Ztschr. anorg. Chem., vol. 83, 1913, p. 39.

[b] Richardson, H. K., Some observations on the laboratory production of aluminum: Trans. Am. Electrochem. Soc., vol. 19, 1911, p. 159; Thompson, M. DeKay, The electrolytic reduction of aluminum as a laboratory experiment: Electrochem. and Met. Ind., vol. 7, 1909, p. 19; Tucker, S. A., The preparation of aluminum in the laboratory: Electrochem. and Met. Ind., vol. 7, 1909, p. 315; Neumann, B., and Olsen, H., Production of aluminum as a laboratory experiment: Electrochem. and Met. Ind., vol. 8, 1910, p. 185: Haber, F., and Geipert, R., Versuche über Aluminumdarstellung: Ztschr. Elektrochem. Bd. 8, 1902, p. 1; Borchers, W., Electric smelting and refining, translated by McMillan, W. G., 1904, p. 148.

[c] British Patent 10057, July 18, 1887.

[d] Plato, W., Erstarrungserscheinungen an anorganischen Salzen und Salzgemischen: Ztschr. phys. Chem. Bd. 58, 1907, pp. 362, 364.

[e] Mierzinski, S., Die Fabrikation des Aluminum und der alkaline Metalle, 1855, p. 56.

[f] Fedotieff, P. P., and Iljinsky, W., Beiträge zur Elektrometallurgie des Aluminums: Ztschr. anorg. Chem., vol. 80, 1913, p. 113.

[g] Ruff, O., and Plata, W., Zur Darstellung des Calciums: Ber. deut. chem. Gesell., Jahrg. 85, 1902, p. 3612. Wöehler, P., Darstellung von metallischen—Calcium für Laboratoriumszwecke; Ztschr. Elektrochem., Bd. 11, 1905, p. 612. Bürgel, C., Über die elektrolytische Gewinnung des Calciums: Ztschr. Elektrochem., Bd 14, 1907, p. 31.

Arndt and Loewenstein[a] show that molten $CaCl_2$ can dissolve 3 per cent SiO_2 at 820° C., 5.5 per cent at 900° C., and 7.5 per cent at 950° C., and that its specific gravity at 800° C. is 2.05 and at 900° C. is 2.00.

Moldenhauer and Anderson[b] give 630° C. as the melting point of the two lowest melting mixtures of $CaCl_2$ and KCl, one consisting of 85 parts $CaCl_2$ and 15 parts KCl, and the other, 40 parts $CaCl_2$ and 60 parts KCl.

It is therefore seen that there are a number of possible mixtures of alkali and alkaline-earth chlorides and fluorides having comparatively low melting points that might serve as a molten cover or flux in melting aluminum chips. Salts of heavy metals are not promising, on account of the reduction of the salts to metal by aluminum, and the salts of few other acids are sufficiently stable at the necessary temperature.

BRIQUETTING BORINGS.

Again it has been suggested that the borings be briquetted, the theory being that in this way the air held by the loose borings is eliminated and the borings put in such shape that they may be readily charged and submerged below the surface of a heel of molten metal.

Such briquets, if made under very heavy pressure, are almost as solid, when cold, as ingot metal. Sperry[c] advocates this treatment, and says—

> In the treatment of aluminum chips this process is particularly important as this metal, more than any other commercial one, is difficult to treat in such a form. When briquetted, the melting would become a simple operation and the resulting metal would be worth using. Metal now made from aluminum chips is of the poorest quality.

It is claimed[d] that borings that gave but 50 per cent recovery when melted loose gave 85 per cent when briquetted, whereas Hirsch[e] states that in melting aluminum borings loose borings were melted in 50 minutes per crucible (capacity not stated), with a loss of 13.8 per cent; but briquetted borings lost only 8.1 per cent and melted in 35 minutes.

Two properties of finely divided aluminum must be taken account of in any successful method of melting chips. First, the readiness with which aluminum oxidizes, and second, the difficulty with which tiny globules of molten aluminum coated with a film of oxide or dirt coalesce.

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[a] Arndt, K., and Loewenstein, W., Über Losungen von Kalk und Kieselsäure in geschmolzenem Chlorcalcium: Ztschr. Elektrochem., Bd. 15, 1909, p. 784.

[b] Moldenhauer, W., and Anderson, J., Über die elektrolytische Darstellung von calcium Legierungen und Calcium; Ztschr. Electrochem., Bd. 19, 1913, p. 444.

[c] Sperry, E. S., Briquetting metal chips: Brass World, vol. 7, 1911, p. 41.

[d] Anon., Briquetting metal turnings and borings: Engineering, vol. 94, 1912, p. 737; Jour. Inst. Metals, vol. 9, 1913, p. 246.

[e] Hirsch, E. F., Metall-briketts: Elektrotech. Ztschr. vol. 35, 1914, p. 1093.

OXIDATION OF ALUMINUM.

Magnesium powder is the main constituent of many photographic flash-light powders; aluminum powder is also largely used for this purpose and, when suitably heated in the air, burns with explosive violence. Because of its rapid oxidation, finely divided aluminum is used in illuminating bombs and range-finding shells for night use in warfare. The affinity of aluminum for oxygen is so great that aluminum powder, when mixed with the finely divided oxide of the metal it is desired to reduce and the mixture heated in one spot by a suitable primer, reacts with great violence, forming molten aluminum oxide and the metal desired. This is the well-known thermit reaction so much used for the production of difficultly reducible metals, and which produces probably the highest temperatures attainable by man, save those of certain electric furnaces.

Finely divided aluminum, mixed with an oxidizing agent such as ammonium nitrate, whose decomposition products are gaseous, has been suggested as an explosive for miners' use.[a]

Aluminum oxide is not reducible to the metal by carbon, even in the electric furnace, only a carbide being formed save in the presence of another metal, as in the Cowles process for the manufacture of aluminum bronze. To reduce the oxide, one must use electrolysis in a fused electrolyte. Aluminum chloride is reducible by metallic sodium or potassium.

Echevarri[b] states that in ordinary melting of ingot the metal should not be heated above 725° C. (1,330° F.), as at that temperature aluminum readily oxidizes with the oxygen of the air.

NITRIDATION OF ALUMINUM.

Not only does the oxygen of the air but also the relatively inert nitrogen combine with finely divided, highly heated aluminum. Several workers[c] find that if aluminum powder is heated in a current of nitrogen to a temperature of about 700° C. the temperature rapidly rises, even to 1,300° C. in the interior of the mass. Matignon[d] states that a temperature of even 2,000° C. is reached, with the formation of aluminum nitride, which coats the tiny globules so that they do not coalesce.

Zappi[e] has noted that the more finely divided metallic aluminum is, the greater the protective effect of the oxide coat, as large pieces of metallic aluminum react with carbon tetrachloride at 100° C., while aluminum powder does not react at 280° C.

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[a] Sesti, G., Italian Patent 133882, Aug. 7, 1913· Chem. Abs., vol, . 1915, p. 1285.

[b] Echevarri, J. T. W., Aluminum and some of its metals used: Jour. Inst. Metals, vol. 1, 1909, p. 128.

[c] Shokoff, I., Aluminum nitride: Jour. Russ. Phys. Chem. Soc., vol. 40, 1908, p. 457; Sci. Abs., vol. 13, 1910, p. 517; Wolf, J., Zur Darstellung von Aluminiumnitride aus den elementen: Ztschr. anorg. Chem., Bd. 83, 1913, p. 159; Fichter, F., Über Aluminiumnitride: Ztschr. anorg. Chem., Bd. 54, 1907, p. 324; Fichter, F., and Spengel, A., Die Reactionen des Aluminiumnitride: Ztschr. anorg. Chem., Bd. 82, 1913, p. 192.

[d] Matignon, C., Synthesis of ammonia by means of aluminum nitride: Jour. Franklin Inst., vol. 178, 1914, p. 794.

[e] Zappi, E. V., Action of metals on chloride of carbon: Anales soc. quim. Argentina, vol. 2, 1914, p. 217; Chem. Abs., vol. 9, 1915, p. 2117.

Vickers[a] states that in melting borings under a heel of molten metal considerable nitrogen is absorbed, which will produce minute pinholes in the castings. He states that 0.5 per cent nitrogen will reduce the strength of aluminum alloy one-half, that it is practically impossible to prevent the absorption of nitrogen in melting turnings, and that the only way to remove the nitrogen is to add titanium. Rossi[b] claims that the addition of titanium to aluminum removes both nitrogen and carbon as titanium nitride and cyanonitride.

Sperry[c] states that aluminum absorbs nitrogen while melting. It is also reported[d] that the higher the temperature to which the metal is heated the more nitrogen is taken up. On the other hand, Fichter[e] states that no nitride is formed in melting large masses of aluminum, even if nitrogen be blown into the melt.

In unpublished experiments of H. S. Bennett, of Cornell University, nitrogen was bubbled through 7 pounds of molten pure aluminum held at 960° C. to 1,010° C. for 70 minutes. Physical tests (including the "hot shortness" test[f]) showed no change in properties, although analysis indicated the presence of traces of nitrogen.

Carpenter and Edwards[g] found a nitrogen content ranging from nothing up to 0.0055 per cent in some of the aluminum used by them. The chief chemist of a large aluminum company states that he has never found over 0.001 per cent. One laboratory states that aluminum may contain from 0.0024 to 0.06 per cent. On two samples of a lot of new ingot metal suspected of containing nitrogen, one laboratory found 0.0571 and 0.0376 per cent, whereas a very careful analyst working on duplicate samples of this lot found only 0.0007 and 0.0004 per cent, and in another lot known to have been very strongly overheated he found 0.0004 per cent.

Inasmuch as aluminum nitride does not appear to be soluble in metallic aluminum, it seems probable that if any nitride really exists in ingot refined from borings, it is mechanically included, although Tchijerski[h] claims that aluminum nitride can form a solid solution with iron. However, nitrogen is absorbed in melting borings or in remelting skimmings, when finely divided metal is present and the mass is heated very hot. A nitrogen content that would correspond to 15 per cent AlN has been found in the dross taken off in the remelting of skimmings, a little $ZnCl_2$ being used as flux.

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[a] Vickers, C., How to melt aluminum turnings and borings: Foundry, vol. 41, 1913, p. 119.

[b] Rossi, A. J., U. S. Patents 1,056,125, 1,085,488, 1,104,371.

[c] Sperry, E. S., Notes: Brass World, vol. 8, 1912, pp. 134, 270.

[d] Anon: Some hints on the melting of aluminum: Castings, vol. 11, 1911, p. 2.

[e] Fichter, F., Über aluminiumnitride: Ztschr. anorg. Chem., Bd. 54, 1907, p. 324.

[f] Norton, A. B., A "hot shortness" testing machine for aluminum alloys: Trans. Am. Inst. Metals, vol. 8, 1914, pp. 124–127.

[g] Carpenter, H. C. H., and Edwards, C. A., Eighth report to the Alloys Research Committee: Proc. Inst. Mech. Eng., 1907, p. 57.

[h] Tchijerski, N. P., Iron and nitrogen: Rev. Soc. Russ. Met., vol. 1, 1913, p. 127; Science Abs., vol. 18, 1915, p. 256; Rev. met., t. 11, 1915, p. 617.

Richards[a] has called attention to the fact that the dross skimmed off of molten aluminum gives off ammonia in moist air, owing to the formation of aluminum nitride.

On the absorption of nitrogen by mixtures of alumina and carbon at high temperatures are based several processes for the fixation of atmospheric nitrogen, such as those of Serpek, who aims to produce aluminum nitride, and of Peacock, who aims to produce a carbonitride. Whatever be the exact product or the reaction that takes place in refining borings, it is certain that both the oxygen and nitrogen of the air enter into the formation of the dross. It is also stated[b] that in melting aluminum in graphite crucibles, if the metal is allowed to become very hot and is then stirred so that particles of graphite are scraped off the crucible, some aluminum carbide will be formed. Whether this be true or not, there is some indication that minute traces of some carbide, whether a carbide of aluminum or of some impurity is not known, may be present in most aluminum, as the odor of a freshly fractured surface, such as that of a tensile test bar, often decidedly reminds one of a faint acetylene-like odor[c] and seems to be stronger on the fracture of metal obtained by remelting borings than on that of ordinary ingot. As aluminum carbide (Al_4C_3) with water produces methane instead of acetylene, it is peculiar that so marked an odor should be present if the carbide is one of aluminum itself.

COALESCENCE OF MOLTEN ALUMINUM GLOBULES.

Whether the globules formed when finely divided aluminum is melted are covered with a thin film of oxide, or mixed oxide and nitride, or possibly carbide, or with fine dirt, they do not coalesce readily, but remain as distinct globules, something like drops of water on a dusty table. If a mass of these coated globules could be pictured the mass would show a honeycomb-like formation with the drops of molten metal as the honey and the film of dirt and oxide as the comb. In other words, there is an emulsion of a solid (the film) and a liquid (the metal). If this emulsion is not entirely broken up and the metal freed from the enveloping film, the whole mass of oxide and dirt with the inclosed metal will be skimmed off from what metal has coalesced in the bottom of the crucible. As soon as air strikes this mass of hot porous dross, as it does when the dross is skimmed off, the tiny metallic globules entangled in the dross oxidize at once. The heat of the reaction is so great that a layer of such dross a few inches thick put on an iron plate a quarter of an inch thick will rapidly burn a hole through the iron.

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[a] Richards, J. W., Aluminum nitride: Trans. Am. Electrochem. Soc., vol. 23, 1913, p. 351.

[b] Anon., Aluminum melting difficulties: Foundry, vol. 42, 1914, p. 73.

[c] See Beckman, J. W., In discussion: Trans. Am. Electrochem. Soc., vol. 19, 1911, p. 177.

Fairly large borings free from dirt form comparatively large globules, which can by their own weight break through the enveloping film of oxide, coalesce well, and melt down without much loss. But the smaller borings, that will pass a 20-mesh sieve and may have a thickness of but 0.005 inch or less, form, it will be seen, almost microscopic globules when melted, the weight of which is not sufficient to break through even the very thin film of oxide, to say nothing of adhering dirt.

Skinner and Chubb[a] state that the oxide film obtained in the electrolytic insulation of aluminum wire which is from 0.0001 to 0.0004 inch thick will stand up without rupturing, in small coils, even when the coil is carrying so much current that the wire is molten within this extremely thin shell.

Seligman[b] and Morrison[c] as well as Schoop[d] state that the film of oxide on untreated aluminum wire, which is, of course, infinitely thinner than the film on the electrolytically insulated wire, prevents two wires from welding together without the use of a flux to dissolve the oxide and give true metallic contact.

In "calorizing" iron or steel—that is, coating it with a film of aluminum and aluminum oxide—the piece to be calorized is packed in a mixture of 5 to 50 parts powdered aluminum, 94 to 49 parts aluminum oxide, and 1 part ammonium chloride and then heated to 900° to 950° C.,[e] or far above the melting point of the aluminum. The globules of aluminum do not coalesce.

ANALOGOUS PROBLEMS IN COALESCENCE OF OTHER METALS.

MERCURY AND TIN.

The failure of the aluminum to coalesce is similar to the behavior of floured or sickened mercury, globules of which when coated with talc, graphite, grease, or other materials of that nature, coalesce with the greatest difficulty.[f] Another case where coalescence of particles of a metal is inhibited by a network of foreign material about them is in fusible tin boiler plugs, where Burgess and Merica[g] find that a little tin oxide may prevent the blowing out of a boiler plug even after the tin is all melted.

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[a] Skinner, C. E., and Chubb, L. W., The electrolytic insulation of aluminum wire: Trans. Am. Electrochem. Soc., vol. 26, 1914, p. 137.

[b] Seligman, R., The welding of aluminum: Jour. Inst. Metals, British, vol. 2, 1909, p. 281.

[c] Morrison, W. M., Discussion: Jour. Inst. Metals, British, vol. 1, 1909, p. 150.

[d] Schoop, M. V., Autogenous welding of aluminum: Electrochem. Met. Ind., vol. 7, 1909, p. 151.

[e] Ruder, W. E., Calorizing metals: Trans. Am. Electrochem. Soc., vol. 27, 1915, p. 254.

[f] See Gowland, W., The metallurgy of the nonferrous metals, 1914, p. 305.

[g] Burgess, G. K., and Merica, P. D., An investigation of fusible tin boiler plugs; Jour. Wash. Acad. Sci., vol. 5, 1915, p. 461. Trans. Am. Inst. Metals, vol. 9, 1915. Adv. copy 3 pp.

BLUE POWDER.

A closer analogy, however, is that of the difficultly meltable "blue powder" obtained in zinc smelting. Blue powder, according to the general consensus of opinion, consists of tiny globules of zinc which, as they condense from the retort, become coated with a thin film consisting mainly of zinc oxide with perhaps some silica.

The presence of an oxide coating on the particles of blue powder is an advantage for one particular purpose, that of sherardizing, since this coating prevents the coalescence of the zinc dust, which is in this case undesirable. Hence, according to Trood,[a] zinc oxide in a zinc dust for sherardizing purposes should not be below 8 per cent.

Roeber[b] distinguishes between physical blue powder, or zinc snow, which is finely divided pure zinc free from any coating and according to Hansen[c] can be easily remelted, and chemical blue powder, which is finely divided zinc coated with oxide and silica and can not be remelted without great loss.

Ingalls,[d] Johnson,[e] Lyon and Keeney,[f] and West,[g] explain the formation of unmeltable blue powder in similar ways.

Peterson[h] states, however, that blue powder has the same chemical analysis as spelter, the difference being merely a physical one, and that attempts to melt blue powder in open vessels fail because of oxidation during the heating, before consolidation can take place.

Bleeker,[i] on the other hand, states that blue powder contains but 80 to 95 per cent of metallic zinc, the rest being oxide, with traces of other impurities as well.

Ingalls[j] gives analyses showing the presence of 7.5 to 10 per cent of zinc oxide, on the average.

By putting blue powder into fused zinc chloride held at a temperature above the melting point of zinc, Bleeker found that some of the zinc coalesced, and that on passing a direct electric current through the molten bath the suspended zinc particles migrated to the cathode and coalesced. The recovery was over 100 per cent, owing to electrolysis of the zinc chloride.

Peterson[k] got promising results by this method. He also tried putting blue powder in an iron cylinder with notches, corresponding to tap holes, in the bottom, placing an iron plunger actuated by a screw on top of the powder, heating the whole to 600° to 700° C., and applying pressure with the plunger. In this way he was able to break the film of oxide and make the zinc globules coalesce, getting a recovery of 80 to 85 per cent.

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[a] Trood, S., Sherardizing: Trans. Am. Inst. Metals, vol. 9, 1915. Advance copy, 11 pp.

[b] Roeber, E. F., Editorial, Met. and Chem. Eng., vol. 10, 1912, p. 451.

[c] Hansen C. A., In discussion, Trans. Am. Electrochem. Soc., vol. 24 1913, p. 138.

[d] Ingalls, W. R., The electric smelting of zinc ore: Met. and Chem. Eng., vol. 10, 1912, p. 482.

[e] Johnson, W. McA., The art of electric zinc smelting: Trans. Am. Electrochem. Soc., vol. 24, 1913, p. 191. Met. and Chem. Eng., vol. 13, 1915, p. 763; U. S. Patent 1,150,271, Aug. 17, 1915.

[f] Lyon, D. A. and Keeney, R. M., Possible applications of the electric furnace to western metallurgy: Trans. Am. Electrochem. Soc., vol. 24, 1913, p. 138.

[g] West, T., Determination of oxygen in copper and brass: Jour. Inst. Met., vol. 10, 1913, p. 379.

[h] Peterson, P. E., The electric zinc furnace: Trans. Am. Electrochem. Soc., vol. 24, 1913, p. 231.

[i] Bleeker, W. F., An electrolytic method for the reduction of blue powder: Trans. Am. Electrochem. Soc., vol. 21, 1912, p. 359.

[j] Ingalls, W. R., The metallurgy of zinc and cadmium, 1903 ed., pp. 205, 529.

[k] Peterson, P. E., Loc. cit.

Peterson's furnace is merely a modification of the old Montefiore furnace for the melting of blue powder, described by Ingalls.[a] This furnace contained upright boot-shaped retorts into which 40 to 50 pounds of blue powder was charged, a clay piston weighted with an iron bar being put on top of the powder in the leg of the boot to supply pressure to break the oxide film. The metal ran out from the toe of the boot. The yield was 85 per cent to 86 per cent, but the metal occluded a good deal of oxide and was hence of poor quality, so that the use of the furnaces was generally given up in favor of redistilling the blue powder.

Cote and Pierron [b] suggest a continuous furnace for the melting of blue powder, in which the powder is fed into a horizontal cylinder within which rotates a solid eccentric cylinder, smooth on one side and bearing helically arranged flanges on the other. The smooth side almost or quite touches the interior of the hollow cylinder and exerts pressure on the mass of blue powder.

The flanges act as a screw conveyor to move the material from the hopper opening at one end to the discharge opening at the other. The blue powder is mixed with a suitable flux and fed into the hollow cylinder, which is kept at 450° to 500° C. (30° to 80° above the melting point of zinc) by various means, among which electrical heating is mentioned, triturated by the inner cylinder and agglomerated into semifluid masses in which the tiny globules have coalesced into much larger ones, and which may therefore be remelted and the oxide released.

It is stated in the patent that from blue powder containing 85 per cent of metallic zinc 80 per cent of the metal, or 68 per cent of the original blue powder, may be recovered, even in a "poor apparatus," and that under good conditions the whole of the metal content may be recovered. As fluxes, sodium, potassium, and calcium chlorides, carnallite, zinc chloride, and sodium and potassium chlorozincates, or mixtures of these, are mentioned. A flux consisting of 60 per cent of NaCl and 40 per cent $CaCl_2$, used in the proportion of 15 to 20 per cent of the weight of the blue powder, is recommended.

No information is at hand as to whether or not this furnace has proved commercially successful or as to the maintenance cost.

Richards[c] states that one of the difficulties in the electrolytic refining of zinc is the deposition of spongy zinc, which loses 20 to 25 per cent on remelting, though this loss is decreased by using sal ammoniac as a flux.

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[a] Ingalls, W. R., The metallurgy of zinc and cadmium, 1903 ed., p. 527.

[b] Cote, E. F., and Pierron, P. R., French Patent 458111, July 29, 1912.

[c] Richards, J. W., The electrolytic deposition of zinc: Trans. Am. Electrochem. Soc., vol. 25, 1914, p. 281.

Thierry[a] states that attempts made to melt blue powder with sal ammoniac result in a loss of zinc and present numerous difficulties so that the method had not been used in commercial practice.

He further states that in order to make the powder coalesce on melting it is desirable to remove the oxide coating by adding just sufficient hydrochloric acid to dissolve the oxide, without leaving any excess acid to act on the metallic zinc, the proper amount of acid being determined by analysis. The cleaned powder is then compressed to remove the solution, and the blocks melted down out of contact with air. Information as to the commercial operation of this process is lacking.

SUMMARY OF METHODS SUGGESTED.

The methods suggested in the literature for melting aluminum chips and for the analagous problem of melting "blue powder" fall into the following general classes:

1. Feeding the chips back into the aluminum reduction pots and subjecting the melt to electrolysis.
2. The use of a flux, or, more strictly, a molten cover, melting at or below the melting point of aluminum or of the alloy to be remelted, in order to exclude air in ordinary furnaces.
3. The use of a true flux that will dissolve aluminum oxide and dirt.
4. Exclusion of air by stirring the chips into a previously molten "heel" of aluminum in an ordinary furnace.
5. Exclusion of air by the use of a vacuum furnace or a retort furnace, or by utilizing the stored heat in the walls of a previously heated furnace.
6. Briquetting the chips while cold in order to press adjacent chips closely together and thus make coalescence more easy, to reduce the amount of air held by the borings, and to permit easy poking of the borings down under the surface of the molten metal.
7. Subjecting a mass of chips to pressure while melting to promote coalescence, or subjecting it both to slight pressure and to constant stirring, as in the puddling method.
8. Promoting coalescence by stirring a volatile material such as NH_4Cl, $ZnCl_2$, or $AlCl_3$, which vaporize at 350°, 730°, and 183° C., respectively, into the mass of globules of metal mixed with oxide and dirt. The gas evolved lifts the particles and stirs them so that some of the tiny globules may come into metallic contact and coalesce. There is also a possibility that the nascent chlorine formed by the dissociation of the substances may react with the oxide film to form the chloride and thus clean the surface of the globule.
9. Cleaning the surface of the chips with chemical solutions before melting.

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[a] Thierry, C. V., U. S. Patent 1030351, June 25, 1912.

METHODS USED COMMERCIALLY IN MELTING CHIPS.

Seligman[a] states that in British practice, as a rule, no flux is used in remelting chips and that the loss is high because of the high temperature needed to free the metal from the dross. He does not consider the use of NH_4Cl or $ZnCl_2$ to be of much value and states that the use of $ZnCl_2$, particularly with pure aluminum, is disadvantageous, as it contaminates the metal with zinc. He has obtained nearly perfect recovery by adding fine scrap and turnings to the reduction pots, but the impurities are likely to upset the bath. He has had good recoveries when using low-melting fluoride-containing fluxes similar to the Schoop flux for welding, even getting ingot from aluminum powder, but such fluxes attack the crucible.

PRACTICE AT AN AMERICAN PLANT.

CRUCIBLE METHOD.

In the practice of one American refining company borings have been melted in various ways, first in coke, pit furnaces, with a small amount of common salt as flux, and later with No. 250 crucibles in either coke or oil-fired pit furnaces. The crucibles were charged with borings and set into the furnace. When the borings became pasty—that is, when the metal melted but had not yet broken through the skin of oxide plus dirt that had formed about each chip—the mass was poked down with a stirrer and more borings added through a long sheet-iron funnel. This was repeated until the crucible was full of molten metal and dross. A little sal ammoniac (commercial ammonium chloride) was added from time to time as the metal was poked and stirred. If the dross seemed free from metallic particles, some of it was taken off with a skimmer during the process of melting, so as to give more room in the crucible. It was considered that the more often the metal was poked down, so as to break the oxide film inclosing the particles, the better would be the results. At the end of the melt the pot was pulled from the fire, and more sal ammoniac was added and stirred and poked well into the dross, a total weight of sal ammoniac equal to about 1 per cent of the weight of borings used being the usual quantity added.

The dross skimmed off in this method was usually somewhat caked together, still contained small globules of molten metal, and was very hot. As soon as it was exposed to the air, violent oxidation, much like the thermit reaction, set in, and the mass glowed with an almost blinding light. If the dross was not spread out thinly, so as to cool rapidly, or else covered thickly with sand, so as to exclude the air, it would get hotter and hotter, evidently from the oxidation and nitridation of the metallic aluminum still contained in it. When a pile of this dross a few inches thick was put on a ¼-inch iron plate, it would melt a hole through the iron in a short time, and the pile would glow for hours. The dross, when put into water or allowed to stand in moist air gave off ammonia.

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[a] Seligman, R., in personal letter to G. M. James, March, 1915.

After the dross had been skimmed from the surface of the metal and any adhering below the surface to the walls or bottom of the crucible had been loosened and skimmed off, the metal was cooled by adding some solid ingot poured from a previous melt, as it was so hot that if poured without cooling the ingot would have a poor surface appearance.

It was necessary to get the metal very hot, as otherwise even the larger globules of metal held by the dross would not be fluid enough to break through the enveloping dross and coalesce.

MELTING IN IRON POTS, WITH PUDDLING AND ZINC CHLORIDE FLUX.

This firm has lately changed from crucible melting to iron-pot melting, the new furnaces being similar to those commonly used in aluminum foundries, where it is now more common to melt aluminum alloys in cast-iron pots than in crucibles. The net metal loss in melting ingots for casting in iron pots is not over one-third of 1 per cent, if reasonable care is taken, when the recovery of metal made on remelting the skimmings from the first melting is taken account of.

The furnace used is cylindrical, lined with fire brick, and covered by a heavy iron plate with a hole in it, into which the pot sets, a flange at the edge of the pot holding it suspended in the furnace. The furnace is not sunk in a pit, but stands about 3½ feet high above the floor. The furnace is fired with oil, the flame from the burner entering the combustion chamber below the pot through a hole in the furnace shell and the waste gases being taken off through a port in the shell near the top. The furnace is usually not tilting, and the metal is ladled out, but tilting furnaces using iron pots have recently come into use for melting ingot. In such furnaces the products of combustion do not sweep over the surface of the metal, as they do in a pit furnace, and the metal loss in melting ingot aluminum alloy is less than in crucibles in a coke fire. Resting on the top of the furnace is a sheet-iron cylinder, with a wide door in the front leading into a conical hood, which takes the fumes from the cutting oil and other impurities and the volatile fluxes off to a short sheet-iron stack.

A few borings are put into the pot and heated, with constant stirring, until they become pasty or semifluid. More borings are then added to the mass, which is not allowed to become really molten, but is kept mushy. The borings are puddled all the time, a furnace tender being required for each furnace. The flame, the stirring, and the feeding of borings are all so regulated that at no time is the metal allowed to become even faintly red.

If the flame is run too high, if the stirring is not sufficient, or if the borings are fed in too fast for proper stirring, oxidation will start in the mass and the temperature will rise rapidly, with consequent increased oxidation and further rise in temperature, till nearly the whole mass will be converted to oxide. Hence the charge is kept small enough—probably between 100 and 150 pounds—so that the furnace tender can keep it all constantly worked over. When no more borings can be added and efficiently stirred in, the pot is covered and the temperature raised somewhat, so as to allow the metal to settle and the dross to rise. Then the bottom and sides of the pot are scraped free from dross and about 2 per cent of fused $ZnCl_2$ is stirred vigorously into the dross on top. After the zinc chloride has volatilized the dross is taken off with a perforated skimmer. Each skimmerful is allowed to remain in the air only the shortest possible time, the dross being at once dropped into an iron tank partly full of water. Much ammonia is evolved as the nitride-bearing dross reaches the water, and some inflammable gases, probably hydrocarbons, are also produced. With this type of furnace it is possible to stir the metal continuously, which was not the case with crucibles in pit furnaces. The vital points of the process lie in keeping the temperature down while the borings are being added and in constant stirring to promote coalescence.

It is difficult to poke borings down into a crucible or to stir them in a pit fire because of the heat, the necessary length of the stirrer handle, and the choking fumes that arise from the oily borings and from the volatile fluxes used at the end of the heat. In the pot method the heat is not great, the workman is only a couple of feet from the pot and can use his stirrer to better advantage, and the fumes are pretty well taken off.

By the pit-furnace method this firm found it difficult to obtain over 50 per cent recovery, whereas with the pot method the recoveries on the original material, not on the metal content, are given as follows: For 1913, 67.8 per cent, for 1914, 68 per cent, and for the first part of 1915, 69.6 per cent. These figures are on material which includes a certain proportion of centrifuged borings. These borings are sent to the refining plant by an automobile company that uses a water-soluble cutting compound, and states that it uses a centrifugal dryer running at 600 revolutions per minute for drying aluminum chips. The dryer takes a charge of about 175 pounds and three or four minutes whirling reduces the liquid content in the borings to 3 to 5 per cent. No water is added to wash out the cutting compound. After the borings are centrifuged they are stored in sheds until a carload has accumulated, when they are sent to the refiner. The average recovery on the dried borings is stated by the company to be 85 to 86 per cent, whereas previous to such drying the recovery on the borings was 50 to 60 per cent. There is little dirt in these borings, as the floors about the machines are kept well scrubbed.

Before the centrifuge was used for drying the borings, if borings wet with the cutting compound were placed in the storage bins after a little time fumes would arise that had an odor similar to ammonia or smoked herring, then the borings would corrode rapidly. Now, there is no loss from corroding or overheated borings. Although the recovery is given by the automobile company as 85 to 86 per cent on the borings as shipped from the plant to the refiner, the refiner states that there is usually 4 to 5 per cent of iron borings mixed with the aluminum. The iron is removed as completely as possible by passing the borings over a magnetic separator. Therefore the recovery on the actual aluminum content of the chips is about 90 per cent.

The statement that an odor as of ammonia or smoked herring arose from the damp borings on standing in the air, heated only by the heat of reaction due to oxidation, indicated an interesting fixation of atmospheric nitrogen at low temperatures. Some of the cutting compound used was obtained and both clean millings of pure aluminum and oily No. 12 alloy chips were soaked with it and allowed to stand in the air, and also were heated somewhat by external means, but no odor of ammonia or amines was noted.

MELTING WITH $ZnCl_2$-NaCl FLUX IN CRUCIBLES.

Another refining plant, which deals but seldom with aluminum chips, reported that it had sprinkled borings with 2 per cent of a strong $ZnCl_2$ solution and added 2 per cent of dry common salt, and run the borings down in crucibles in coke fires, getting a recovery of 80 to 82 per cent, but the borings were very clean and nearly free from oil or dirt.

Another refining company which uses a reverberatory furnace is said to get a recovery of 80 per cent. The process is held secret and no details are available as to the exact method of melting, the fluxes used, if any, or as to the quality of borings on which an 80 per cent recovery is made.

REMELTING DROSS.

One aluminum foundry which used merely to remelt the skimmings taken from the regular melting pots and to sell the dross from the remelting at $20 per ton now washes them in a wet tumbling barrel, obtaining a concentrate of 800 to 900 pounds of large buttons, plus the adhering dross, from a ton of original dross. The concentrate is remelted promptly—as if left in the air it would oxidize and heat—and 400 to 500 pounds of ingot, worth $60 to $250, depending on the price of aluminum, is obtained, the saving being far greater than the cost of treatment. The melting is done in iron pots and sal ammoniac is used as flux. The average recovery is only about 60 per cent of the actual metal content of the concentrate.

In melting down aluminum grindings from the emery wheels used in grinding the rough castings, a recovery of about 6 per cent on the total weight of aluminum plus dirt (the dirt forming at least half the weight) was obtained by this firm, using $ZnCl_2$ as flux. The grindings as they come from the wheels ordinarily sell for one-half to 1 cent per pound.

This firm made a few experiments on melting borings by heating an iron pot very hot, putting $ZnCl_2$ in the bottom and immediately charging all the borings the pot would hold, then heating very rapidly, the idea being to melt rapidly and to stir the whole mass by the rising vapor from the $ZnCl_2$. Definite figures on this method are not available, but it is stated not to have been very satisfactory.

MELTING WITH SALT AND FLUORSPAR IN CRUCIBLE COKE FURNACE.

At another large refining plant the borings are passed through a magnetic separator and then over an inclined, vibrating, fine-meshed Newago screen, the screen being continually struck by a set of automatically operated small hammers to keep the openings from clogging. The borings are purchased by weight after as much as possible of the iron and dirt is thus removed. This firm has also experimented with various jigs, shakers, and other mechanical devices for separating brass borings from the aluminum borings in the dry way by means of the difference in specific gravities, and with liquation devices for removing pieces of babbitt and white metal, apparently without very satisfactory results.

Passing the chips over a cloth through which air is blown from below, in order to blow out the fine dirt, has also been tried by this firm, but is apparently not so satisfactory as the screen.

The borings are intimately mixed with quite large proportions, apparently between 15 and 30 per cent, of a flux consisting of common salt with a small percentage, probably about 15 per cent, of fluorspar. The mixture of borings and flux is charged into crucibles of about No. 120 size and heated in coke fires. More mixture is added as soon as the first charge can be packed down to make room for it, until the crucible is full. Little stirring, aside from this packing, is done till the end of the heat. The temperature is raised till the flux is fluid enough for the metal globules to separate from it well, when the melt is stirred to promote coalescence of the globules, skimmed, and poured. The flux and dross skimmed off are allowed to cool, crushed, leached with water, and any metallic buttons are recovered by various means of mechanical concentration. The chemist of this firm states that he considers the problem of melting borings as strictly analagous to welding, for which a flux that will actually dissolve aluminum oxide and dirt, such as the fluxes made up of alkali chlorides and fluorides, are essential. He chose a salt and fluorspar flux for commercial use because of its cheapness. On perfectly clean borings, of good size, recoveries of 95 to 98 per cent are reported by this method, but it is stated that with very oily and dirty borings, where a good deal of fine dirt, such as floor sweepings, foundry sand, or road dust, the latter sometimes intentionally added by unscrupulous junk dealers, adheres to the borings, the recovery falls to 65 per cent.

This firm occasionally uses the same flux, but melts in a reverberatory furnace heated very hot before being charged, as much of the metal as possible being melted by the heat stored in the walls, but ordinarily prefers the crucible method, although the recovery of metal from the reverberatory process is said to be almost if not quite as good as from crucibles.

EXPERIMENTS IN MELTING ALUMINUM CHIPS.

GENERAL METHOD OF MAKING TESTS.

In order to compare various methods of melting on the same lot of chips, two barrels of "No. 12" (92 per cent Al, 8 per cent Cu) chips were obtained. These chips were from the milling machines and drills of a large automobile manufacturing company, and were thought by the firm supplying them to be of such quality that on commercial melting in iron pots, by the puddling method, the recovery would be about 50 to 65 per cent, as that was the recovery usually obtained by a refining company using that method, on the regular run of borings from this automobile firm.

The tests were necessarily on a small scale, a charge of 1 to 2 kilograms being used, in a small crucible heated in a gas-fired furnace.

The metal was poured into small ingot, the relative weight of which to the original weight of borings is recorded as the "primary" yield in the tables of results. The dross, when possible, was skimmed into water, but if the nature of the flux made this procedure dangerous, the dross was allowed to cool, was crushed, and washed in water. The wet dross, with the admixed buttons of metal, was washed in a stream of water on a 20-mesh riddle. The buttons, if large and clean, were weighed without remelting; if small and with adhering dross, they were remelted with the same flux and in the same manner as the original melt. The large, clean buttons, or the ingot recovered from the smaller buttons, form the "secondary" yield. The sum of the primary and secondary yields is the total yield on the basis of original weight, that is, on the weight of borings plus oil and dirt. The total yield on the basis of metal content is obtained by dividing the total yield on the basis of original weight by the metal content of the dirty borings.

KIND OF CHIPS USED.

The chips in barrel A contained about 3 per cent of oil and over 7 per cent of very fine dirt. After the oil had been extracted with gasoline the material was screened; 72 per cent remained on a 20-mesh screen, 18 per cent was between 20 and 80 mesh, and 7 per cent passed the 80-mesh screen. Microscopic examination of the material passing the 80-mesh screen showed only traces of aluminum, the vast bulk of it being merely dirt, mainly siliceous, and appeared to be sweepings from a cement floor. Sticks, paper, peach pits, oily waste, chewing tobacco, and apple cores were present in both barrels. All of the dirt could not be sifted out, even after the oil had been extracted with gasoline. The metal content of barrel A was something less than 90 per cent, but 90 per cent is taken in calculating the total yield on the metallic basis. On material from which the oil had not been extracted, so that the fine borings stuck to the larger ones, sieve tests of borings from barrel A gave the following results:

Over 20 mesh, 80 per cent, which equals 76 – per cent of metal plus 4 + per cent of oil and dirt; passing 20 mesh, 20 per cent, which equals 14 – per cent of metal plus 6 + per cent of oil and dirt, figured on weight of original material, or the oily borings over 20 mesh contained about 95 per cent of metal, whereas those passing the 20-mesh screen contained about 70 per cent of metal.

When borings from lot A from which the oil had not been extracted were sized on 14 and 40-mesh riddles the results were: 54 per cent over 14 mesh, 42 per cent between 14 and 40 mesh, 4 per cent under 40 mesh.

The chips in barrel B contained about 2 per cent oil, and on chips from which the oil had not been extracted a sieve test showed the following results: 57.5 per cent over 14 mesh, 30.3 per cent between 14 and 40 mesh, 12.2 per cent under 40 mesh.

The material under 40 mesh was practically all dirt, only traces of metallic aluminum being found in it. As about 5 per cent of oil and dirt could be separated from the material over 40 mesh in size, by extraction with gasoline and sifting on an 80-mesh sieve, the total nonmetal content was about 17 per cent. The metal content of barrel B, for purposes of computation, is taken as 85 per cent. The contents of both barrels had been passed through a magnetic separator, but the removal of iron was far from complete.

CONTROL OF TEMPERATURE.

Control of the temperature was attempted, an iron-constantin thermocouple of the rod and tube type being used, but the temperatures were found to vary throughout the charge. The upper part of the charge would become much hotter than the lower part when any oxidation was taking place in the layer of dross and globules of metal overlying the coalesced mass of metal, as when the furnace cover was removed just before taking out the crucible and pouring. With fluxes that prevented any visible oxidation, the temperature at the bottom of the melt was often higher than that of the flux on top, because of the poor heat conduction in a heavy layer of molten flux. In the results of experiments where volatile fluxes were used the temperatures given are those of the upper part of the pool of coalesced metal, taken just below the overlying layer of dross, at the time the crucible was taken from the furnace; when other fluxes were used the temperatures given are those of the layer of flux.

MELTING WITH VOLATILE FLUXES.

The results of a series of experiments in which aluminum borings were melted with various volatile fluxes were as follows:

Results of melting borings with volatile fluxes.

NO FLUX USED.

Run No.	Chips from barrel—	Observer.	Weight of chips used.	Flux used, percentage of weight of chips.	Approximate final temperature.	Yield of metal.[a]		
						Primary yield, based on weight of chips.	Total yield, based on weight of chips.	Total yield, on basis of metal content of chips.
			Kilogram.		*°C.*	*Per cent.*	*Per cent.*	*Per cent.*
1	A	G	2		910	41	49	54.5
NH$_4$Cl FLUX USED.								
2	A	G	2	2	800	62.5	70	[b] 77
3	A	G	2	2	725	45	60.5	67
4	A	G	2	5	800	59	65.5	72.5
5	A	G	1	2	850	54	60.5	67
6	A	G	1	10	850	61	64.5	72
7	A	J	1	3		46	49.5	[b] 55
Average							62.5	69.5
ANHYDROUS ZnCl$_2$ FLUX USED.								
8	A	G	2	2	875	61	66.5	[b] 74
9	A	G	2	2	760	46	62	69
10	A	J	1	3	800	15.5	48.5	[b] 54
Average							59	66
ANHYDROUS AlCl$_3$ FLUX USED.								
11	A	G	2	2	850	53.5	65	72

[a] The yields are figured only to the nearest one-half per cent.

[b] The borings were not of uniform size in different parts of the barrel, and were difficult to mix thoroughly. Runs 2 and 8 were companion runs on a part that consisted mostly of borings larger than the average, and runs 7 and 10 were companion runs on some borings smaller than the average. The runs were not made in the order given in the table.

In all of these runs borings were packed tightly into the previously hot crucible, poked down as fast as possible, and more borings added with frequent poking and stirring till the whole charge was in. The charge was then stirred from time to time until the end of the melt, when the caked dross that had collected on the sides and bottom of the crucible below the surface of the pool of metal was scraped up, the fluxes were stirred vigorously into the dross, the crucible taken from the furnace, the dross skimmed into water, and the metal poured. If the prices of the three volatile fluxes used be taken into consideration there is not much choice among them on the basis of these results. On a larger scale, the zinc chloride would probably be preferable, as it does not decompose at so low a temperature as the other fluxes, and hence can be stirred into a large mass of dross without much of it volatilizing before it can be stirred in.

MELTING IN ATMOSPHERE OF CHLORINE-CONTAINING GASES.

As the effect of NH_4Cl, $ZnCl_2$, and $AlCl_3$ might be due to the chlorine given off in their decomposition, melting chips in an atmosphere of chlorine or of dry hydrochloric acid gas was tried. The results were as follows:

Results of melting in presence of chlorine and hydrochloric acid gases.

	Run No. 12.	Run No. 13.
Barrel from which chips were taken	A	A
Observer	G	G
Weight of chips used, kilograms	2	2
Gas used	Cl_2	HCl
Approximate final temperature, °C	810	825
Yield of metal:		
Primary yield, percentage of weight of chips	41	32.5
Total yield, percentage of weight of chips	56	56
Total yield, percentage of metal content of charge	62	62

In run 12, Cl_2 gas from a cylinder was run into the top of the crucible, which was open, and the charge was poked and stirred as in runs 1 to 11. In run 13, dry HCl gas was led in through a hole in the crucible cover and the crucible was opened only to poke the charge down and add more borings. Some $AlCl_3$ was formed in this run.

MELTING WITH CONTINUOUS PUDDLING.

After it was found, late in the investigation, what great stress the the refining company that used the iron-pot method with continuous stirring or puddling laid on keeping the temperature down and on the necessity of continuous stirring, that method was tried on chips from barrel B, as the contents of barrel A had been used up. The results were as follows:

Results of melting with continuous puddling.

	Run No. 14.	Run No. 15.
Barrel from which chips were taken	B.	B.
Observer	G., J.	J.
Weight of chips used, kilograms	1.5	1.5
Flux used	$ZnCl_2$.	$ZnCl_2$.
Quantity of flux used, percentage of weight of chips	2	2
Approximate final temperature, °C	800	800
Yield of metal:		
Primary yield, percentage of weight of chips	63.5	54
Total yield, percentage of weight of chips	66	60
Total yield, percentage of metal content of charge	78	71

The temperature was not allowed to rise above 700° C. during the addition of borings. As the furnace was not of the iron-pot type, but a crucible was used setting loosely in the furnace, it was difficult for one observer to stir the pasty mass properly and at the same time hold the crucible in place. The gases of combustion also passed over the crucible, which is not the case in the iron-pot furnace, and hence, as the furnace cover had to be left off to permit stirring and holding the crucible, it was more difficult to prevent oxidation than in the regular iron pot. Run 14 was more thoroughly stirred than run 15.

RETORTING.

In one run the conditions of retorting were approximated by packing 1 kilogram of borings into a hot crucible large enough to contain all the borings at the start. The crucible was then tightly covered and heated without any stirring or poking till the end of the melt, when sal ammoniac was added and the mass stirred and skimmed. The results were as follows:

Results of retorting aluminum chips (run No. 16).

Sample used, chips from barrel B.
Observer, G.
Weight of chips used, 1 kilogram.
Flux used, $ZnCl_2$.
Quantity of flux used, on basis of weight of chips, 2 per cent.
Approximate final temperature, 900° C.
Yield of metal:
  Primary yield, on basis of weight of chips, 58 per cent.
  Total yield, on basis of weight of chips, 64 per cent.
  Total yield, on basis of metal content of charge, 71.5 per cent.

MELTING IN A REDUCING ATMOSPHERE.

In order to try the effect of melting aluminum scrap in a strongly reducing atmosphere some tests (Nos. 17 to 21) were made in an electric crucible lift-out furnace of the Hoskins type and some (Nos. 22 and 23) in one of the indirect-arc type, in both of which furnaces gas analyses showed the atmosphere to be strongly reducing when the furnace was closed. These tests were made before the others herein recorded, on a sample of borings (sample C) that were not so dirty as those from barrels A and B. Sample C contained more oil than samples A and B, however. It was estimated that sample C contained about 5 per cent oil and not over 5 per cent dirt. The dross in runs 17 to 23 was not skimmed into water, and the buttons held in it were oxidized completely, so that the results of the tests, which give only the primary yield, would have been increased somewhat by the secondary yield had the buttons in the dross been recovered. The results follow:

Results of melting borings in a reducing atmosphere.

Run No.	Sample—	Observer.	Weight of chips used.	Flux used.	Amount of flux used, percentage of weight of chips.	Approximate final temperature.	Furnace used.	Primary yield, percentage of weight of chips.
			Pounds.			*°C.*		
17	C.	G. and L.	12.5	NH_4Cl	2	800	Crucible	40
18	C.	...do	14	...do	2	760	...do	55
19	C.	...do	29	...do	2		...do	65.5
20	C.	...do	24.5	...do	2		...do	63
21	C.	...do	28	...do	2	700	...do	52
22	C.	...do	40	...do	2		Arc	30
23	C.	...do	30	...do	2		...do	50

The melts in the crucible furnace were poked and stirred quite a good deal while the oil contained in the borings was smoking off, as the furnace cover could be opened without danger of getting an oxidizing atmosphere, but thereafter no stirring was done till the final addition of NH_4Cl just before taking out the crucible. In the runs in the arc furnace no stirring was done, and the furnace door was opened as little as possible in charging. In the arc furnace the local overheating of the borings as soon as charged seemed to cause enough oxidation, through the air held by the borings, to prevent coalescence, so that a large proportion of dross was formed, which entangled much metal.

HEATING IN ABSENCE OF AIR.

In melting borings from sample A in the gas furnace 1 kilogram of borings was rammed down into the bottom of a hot crucible, and 1 kilogram of molten metal at 800° C., from another crucible, was poured on top of them so as to cover them completely and then the whole was heated. The vapor from the oil in the borings bubbled up through the molten metal that remained on top. No stirring was done till a thermocouple plunged to the bottom of the crucible showed a temperature of 800° C., when the whole was stirred and much dross that had collected on the sides and bottom of the crucible was brought to the top. NH_4Cl was then stirred in and the crucible skimmed into water. The results of the test were as follows:

Results of melting borings in absence of air (run No. 24).

Sample used, chips from barrel A.
Observer, G.
Weight of chips used, 1 kilogram.
Flux used, NH_4Cl.
Quantity of flux used, 2 per cent.
Approximate final temperature, 800° C.
Yield of metal:
  Primary yield, on basis of weight of chips, 50 per cent.
  Total yield, on basis of weight of chips, 61 per cent.
  Total yield, on basis of metal content of charge, 68 per cent.

USING A HEEL OF MOLTEN METAL.

Another run was started with a "heel" of 1 kilogram of molten metal in the bottom of the crucible. One kilogram of borings was added a little at a time, an attempt being made to poke them down continually below the surface of the fluid metal—that is, to follow the usual directions for using a heel of metal. This procedure differs from the puddling process in that there is fluid metal in the bottom of the pot, whereas in puddling the whole mass is kept pasty during the addition of borings.

The chips floated on the metal and obstinately refused to stay submerged. The temperature was kept rather low throughout and was, as it proved, not raised sufficiently at the end to give a good separation of metal from dross. The results were as follows:

Results of melting chips with heel of molten metal (run No. 25).

Sample used, chips from barrel A.
Observer, G.
Weight of chips used, 1 kilogram.
Flux used, NH_4Cl.
Quantity of flux used, on basis of weight of chips, 2 per cent.
Approximate final temperature, 700° C.
Yield of metal:
  Primary yield, on basis of weight of chips, 39 per cent.
  Total yield, on basis of weight of chips, 59.5 per cent.
  Total yield, on basis of metal content of charge, 66 per cent.

Other tests on this method and some of the other methods described will be given under the results of sizing and briquetting the chips.

CONCLUSIONS AS TO RESULTS OF TESTS.

In all of the tests described in the preceding pages the fluxes used were volatile and did not form any slag that would tend to protect the metal from oxidation; the dross skimmed off was voluminous, loose, and contained many tiny metallic globules. If any considerable mass of this dross is left in the air it becomes white hot, and then cakes, the loose particles of dirt and oxide being compactly cemented together by oxide formed from the oxidation of the metal in the buttons. No metallic buttons will be found in the mass after cooling in air unless the globule was originally at least the size of a marble and the coating of oxide about it happens to have been so impervious to air that the inside of the globule is protected. Each tiny globule becomes oxidized throughout. The cooled dross is gray in the mass from the presence of dirt, but many tiny globular white specks of aluminum oxide, pseudomorphic after the metal, show where tiny globules of molten metal were contained in the dross when skimmed off.

By showering the hot dross with its entangled globules of metal through the air slowly one can produce an almost continuous sheet of flame. It is impossible to skim the dross into water quickly enough to prevent the burning up of the smallest globules, as exposure for a fraction of a second to the air is sufficient for their oxidation and nitridation. When any considerable amount of dross is skimmed into water there is a very strong odor of ammonia mixed with hydrocarbons. The gas bubbles that arise from the hot dross as it touches the water take fire spontaneously. It appears that there is a good deal of nitrogen and a little carbon chemically combined with the dross.

Although in the last analysis the loss of metal in these methods of melting borings is due to oxidation and nitridation, yet very little of this loss takes place within the furnace in commercial practice in melting in a pit furnace or in an iron-pot furnace, if the pot is covered at the end, or in the gas furnace used in the tests. The dross does not begin to show the thermit-like glowing until it comes in contact with the air. If each and every one of the tiny globules could be made to coalesce in the crucible and to free itself entirely from the dirt and from the small quantity of oxide that actually forms in the furnace, the loss would be very small. The root of the trouble lies in the failure of the tiny, light globules to coalesce.

With this premise it becomes plain why the presence of fine dirt makes the recoveries low, and why, with a given amount of dirt, the smaller the chips are the lower is the recovery. The greater the amount of dirt and the more finely divided the dirt is, the greater is its power to form the honeycomblike coating which envelops the globules and prevents their uniting. The tinier the chip the greater is the surface of the globule in respect to its weight. The less the weight of the globule the less is the force tending to tear through the enveloping film.

When the borings are even slightly oily, any dust in the borings sticks tightly to them and a surface film of dust tends to be formed before the chip is melted that under the effect of surface tension readily forms a bag about the tiny drop into which the chip changes on fusion.

The small size of some of the chips can hardly be prevented, as they come from the light finishing cut, but the content of dirt can be greatly reduced if care is taken in the collection of the chips.

The tendency toward oxidation of fine chips can be more easily and more readily checked in melting than their reluctance to coalesce can be overcome. It does not seem that, without the use of some slaglike flux to act as a collector of the tiny buttons, such methods as retorting, vacuum-furnace melting, etc., which aim at preventing oxidation, but do not provide for stirring, or other means of promoting coalescence, are likely to offer any advantage in the melting of dirty chips, at least, commensurate with their cost and the difficulties of their operation.

It would therefore appear that if some menstruum or slag were present through which the globules would drop, they might coalesce better, as the dirt would tend to be soaked up by the slag. There would no longer be a honeycomblike coating of solid dirt enveloping the globules, but the globules and the dirt would both be dispersed in a liquid slag. The slag, if lighter than the metal, would also prevent access of air. Hence it seems possible that a molten cover, not necessarily acting as a solvent of dirt and oxide, but merely soaking them up and taking them away from the globules might both promote coalescence and prevent oxidation. It is plain that the most efficient menstruum will be one which is fluid below the melting point of the metal, and which has not only a lower specific gravity than the metal, but also has a low viscosity, so as to allow the globules to sink readily through it.

MELTING WITH MOLTEN COVER.

Suitable molten covers combining these properties with cheapness and with chemical inertness to the metal are, however, not easy to find. Alkali and alkaline-earth chlorides, or mixtures thereof, form the basis of most of the molten covers suggested.

TESTS WITH A COMMERCIAL FLUX.

One such flux is on the market; it has been mentioned, with the makers' directions for using it in melting ingot, on page 17. Although some aluminum founders who have tried this flux reported unfavorably on its use for melting ingot, one even denouncing it as a fraud, some of it was obtained and tried on borings. The flux is a lavender-colored granular material, is soluble in water, tastes like potassium chloride, and melts at about 620° C. The lavender color is due to some organic dye, as it may be extracted by absolute alcohol, in which the material itself is not soluble. The color burns off on ignition, before the melting point is reached. It is perhaps added for psychological effect on the purchaser. Qualitative analysis shows the presence of K, Na, Cl, SO_4, and a trace of Mg. Quantitative analysis was not made, but it appears that a molecular mixture of KCl and NaCl, plus a little K_2SO_4 would give a mixture of about the same melting point and properties.

In all the following tests on molten covers and other fluxes, the powdered flux was mixed with the borings before charging, and the dross or slag, except where so stated in the tables, was not skimmed directly into water, on account of the explosions that occur in such a case. The dross or slag mixed with dross was skimmed off, allowed to cool somewhat, and thrown into water while still hot in order to disintegrate it. It was then crushed, washed on a 20-mesh sieve, and hand picked. If only a few large clean buttons were obtained, their weight was added to the weight of ingot to give the total yield. If the buttons were small and adhered to particles of slag or dross, they were remelted with the same proportion of the same kind of flux as was used in the original melt and the weight of secondary ingot thus obtained was added to that of the original ingot to give the total yield.

The results of the test with the commercial flux mentioned were as follows:

Results of tests with commercial flux.

Run No.	Chips from barrel—	Observer.	Charge used.	Flux used, percentage of weight of chips.	Approximate final temperature.	Yield of metal.		
						Primary yield, on basis of weight of chips.	Total yield, on basis of weight of chips.	Total yield, on basis of metal content of charge.
			Kg.		*°C.*	*Per cent.*	*Per cent.*	*Per cent.*
26	A	G	2	5	850	45.0	58.0	64.0
27	A	G	2	25	760	48.5	62.0	69.0
28	B	J	1	25	900	55.0	62.5	73.5

TESTS WITH DEHYDRATED CARNALLITE.

Dehydrated carnallite, or a mixture in molecular proportions of KCl and $MgCl_2$ has a low melting point and has been suggested as a flux. Tests with this flux gave the following results:

Results of melting borings with flux of dehydrated carnallite.

	Run No. 29.	Run No. 30.
Barrel from which chips were taken	A	A
Observer	G	G
Weight of chips used, kilograms	2	2
Percentage of flux used, on basis of weight of chips	5	25
Approximate final temperature, °C	725	800
Yield of metal:		
Primary yield, percentage of weight of chips	47	48
Total yield, percentage of weight of chips	60.5	59
Total yield, percentage of metal content of charge	67	65.5

DISCUSSION OF RESULTS WITH THE TWO FLUXES.

Neither flux, even when 25 per cent was used, was in sufficient quantity to soak up all the dirt and dross and still become truly fluid. Neither flux rose entirely to the top, but much stuck to the sides and bottom of the crucible.

SPRINKLING WITH ZINC-CHLORIDE SOLUTION AND DRY SALT.

As the use of 2 per cent of a strong solution of $ZnCl_2$ and 2 per cent dry NaCl sprinkled over the borings had been suggested (see p. 30), this was tried. The results of the test were as follows:

Results of melting borings sprinkled with $ZnCl_2$ solution and dry NaCl (run No. 31).

Barrel from which chips were taken, A.
Observer, J.
Weight of chips used, 1.5 kilograms.
Quantity of flux used, 2 per cent of $ZnCl_2$ solution, 2 per cent of dry NaCl.
Approximate final temperature, 1000° C.
Yield of metal:
  Primary yield, on basis of weight of chips, 45 per cent.
  Total yield, on basis of weight of chips, 58 per cent.
  Total yield, on basis of metal content of chips, 64 per cent.

The slag was very sticky, almost solid with the dross and dirt soaked up by it, so that it was necessary to heat it very hot to get any separation of metal from it. The slag and dross showed a thermit-like reaction when exposed to air.

TESTS WITH COMMON SALT.

As common salt has been used in commercial practice and is the cheapest alkali chloride, a series of tests was made with varying proportions of an "agricultural grade" of salt, costing $2 per ton at the salt works, as flux. The temperatures given are the temperatures in the slag layer at the end of the heat, not in the metal. The results of the tests follow:

Results of melting borings with common salt.

Run No.	Chips from barrel—	Observer.	Charge used.	Flux used, percentage of weight of chips.	Approximate final temperature.	Yield of metal.		
						Primary yield, on basis of weight of chips.	Total yield, on basis of weight of chips.	Total yield, on basis of metal content of charge.
			Kg.		*°C.*	*Per cent.*	*Per cent.*	*Per cent.*
32	A	J	1	15	900	45.5	62	69
33	A	J	2	20	900	66	69.5	77
34	A	J	1	25	900	48.5	61	68
35	A	G	1.5	50	900	66	76.5	85
36	A	J	1	50	900	59	71	79
37	A	J	1	50	900	54.5	61.5	68.5
38	A	J	1	50	900	59	65	72
39	A	J	1	100	900	47	66	73

With as little as 15 per cent salt the dross was not all soaked up and the mass was dry in places and not wholly pasty and the thermit-like reaction was not wholly stopped. The separation was not good. With 100 per cent of flux the volume of slag was too great to give good separation; 20 to 50 per cent of salt gave pasty slags that were easily skimmed off, gave fair separation, and prevented the thermit-like reaction, although there was not perfect coalescence, as vast numbers of tiny buttons were still held in the slag, passed the 20-mesh sieve, and were lost.

TESTS BY A FOUNDRY WITH SALT AND CALCIUM CHLORIDE FLUX.

Some tests by a foundry, reported to the writers, on NaCl, $CaCl_2$, and a mixure of the two, as flux, are given below.

SERIES 1, MELTING EMERY GRINDINGS.

Some experimental melts were made in a small crucible, oil-fired furnace; the material used was sawdust from band saws used in cutting off gates, etc., plus emery grindings and emery dust from the grinding wheels. The probable metallic content was between 60 and 70 per cent, the rest being emery dust, floor sweepings, and paraffin used as a lubricant in sawing and grinding. The material would nearly all pass a 20-mesh sieve. The results were as follows:

Charge used.—1¾ kilograms.

Run A.—No flux was used. The total recovery of metal was 19 per cent on original weight, all as ingot, no metal being recovered from the dross, as this burnt up on removal.

Run B.—In this run 75 per cent NaCl was used as flux. The total recovery of metal was 47 per cent. Much of this metal was from buttons in the dross, which was not fluid, but was very pasty, almost dry.

Run C.—In this run 30 per cent commercial fused $CaCl_2$ flux was used. The flux melted and dust was poked constantly into it. The recovery was 43 per cent in good ingot.

Run D.—In this run 75 per cent flux, which was two-thirds commercial fused $CaCl_2$, one-third NaCl, was used. The total recovery was 38 per cent, mostly in ingot; some metal was recovered as large buttons in the flux cover.

Run E.—This run was made on 60 per cent of old flux, from run D, containing a few aluminum shots from that charge which were not picked out. The recovery was 44 per cent, but most of the metal was buttons from the flux, which was not sufficiently fluid on re-using.

SERIES 2, MELTING DIRTY BORINGS WITH SAL AMMONIAC AND SODIUM CHLORIDE—CALCIUM CHLORIDE FLUXES.

A comparison of sal ammoniac and the $NaCl-CaCl_2$ mixture as fluxes was made. Exceptionally small, thin, oily, and dirty borings, of unknown metallic content, of very poor quality, were melted in 100-pound charges in an oil-fired iron pot.

Run A.—The borings were constantly poked, and 1 per cent sal ammoniac was used at the end of the melt. The recovery was 12½ per cent.

Run B.—The borings were mixed with 20 per cent of the $CaCl_2-NaCl$ flux, and were poked occasionally. The recovery was 42 per cent.

SERIES 3, TESTS ON CLEANER BORINGS WITH SAL AMMONIAC AND SODIUM CHLORIDE—CALCIUM CHLORIDE FLUXES.

A similar comparison of the two fluxes was made on commercial borings, of much better quality than in series 2, but containing some oil and dirt. The borings were melted in crucibles in coke fires.

Charges aggregating 316 pounds were melted, with 1 per cent of sal ammoniac as flux. The recovery was 144 pounds, or 44 per cent. On other charges aggregating 346 pounds, with 26 pounds of fused commercial $CaCl_2$ and 16 pounds NaCl, the recovery was 194 pounds, or 56 per cent.

The flux did not meet with favor by those running the remelting work, largely because of its hygroscopic nature and the consequent difficulty of avoiding dangerous explosions when it is used.

TEST WITH MIXTURE OF CALCIUM CHLORIDE AND SODIUM CHLORIDE.

Chips from lot A were melted with 50 per cent of a flux consisting of 90 per cent commercial fused $CaCl_2$, which contained quite a proportion of NaCl, and 10 per cent added NaCl, the mixture becoming fluid at 600° C. The results of the tests were as follows:

Results of melting borings with flux of $CaCl_2$ and NaCl (run No. 40).

Barrel from which chips were taken, A.
Observer, G.
Weight of chips used, 1.5 kilograms.
Quantity of flux used, 50 per cent (90 parts $CaCl_2$, 10 parts NaCl).
Approximate final temperature, 900° C.
Yield of metal:
  Primary yield on basis of weight of chips, 61 per cent.
  Total yield on basis of weight of chips, 72 per cent.
  Total yield on basis of metal content of chips, 80 per cent.

The flux, though fluid at 600° C., was only just thin enough to skim off properly at 900° C. because of the dirt soaked up by it.

FLUXES CONTAINING FLUORIDES.

In order to get a flux that will not only act as a molten cover to protect the metal from oxidation and a collector to soak up the dirt and permit the globules to coalesce, but will also have a true fluxing or dissolving action both on the dirt and the aluminum oxide, the use of fluorides, as suggested by Schoop,[a] was tried.

TESTS WITH VARIOUS FLUORIDE FLUXES.

Various fluoride fluxes were first used, to find out whether better recoveries were obtainable than with the molten covers Flux No. 1 was as follows: Fluorspar (CaF_2) 16 parts, cryolite (AlF_3–NaF) 28 parts, aluminum fluoride (AlF_3) 56 parts, this being, according to Wright,[b] the composition of the electrolyte used in the production of aluminum. The constituents were fused together, cooled, and the mass pulverized.

Plato[c] shows that a mixture of about 86 parts $CaCl_2$ and 14 parts CaF_2 melts at about 650° C.

Such a mixture (flux No. 2) was made, fused, and pulverized. It was fairly fluid at about 700° C, and the CaF_2 seemed to be all dissolved.

Gleason[d] suggests a flux of fluorspar and anhydrous boric acid, in the proportions of three parts of fluorspar to one of boric acid, for another purpose, that of introducing boron into copper by treating such a flux with aluminum and adding it to copper. It was thought that this mixture might give a fluoride flux for the purpose in question, but it was not sufficiently fluid, even at 1100° C., so it was not used.

Jones[e] suggests a flux consisting of equal parts $LiCl_2$, KCl, and NaF, for use in welding aluminum. Such a flux (No. 3) was made except that KF was used instead of NaF, no NaF being at hand, then fused and pulverized. This flux was extremely fluid at a low red heat.

A flux consisting of 10 parts Na_2CO_3, 2 parts K_2CO_3, 2 parts cryolite, and 1 part borax has been suggested[f] to be used in the proportion of 1½ pounds per hundredweight of chips. The content of fluorine is so small that this can scarcely be classed as a fluoride flux. Such a flux (No. 4) was made, fused, and pulverized.

Powdered cryolite flux (No. 5) was also tried.

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[a] Schoop, M. V., U. S. Patent 943,164, Dec. 19, 1909.

[b] Wright, J., Electric furnaces and their industrial applications, 1908, pp. 186–187.

[c] Plato, W., Erstarrungs erscheinungen an anorganischen Salzen und Salzgemischen: Ztschr. phys. Chem. Bd. 58, 1907, p. 362.

[d] Gleason, E. D., U. S. Patent 1,076,973.

[e] Jones, J. L., Shop problems—fluxing: Metal Ind., vol. 11, 1913, p. 528.

[f] Anon, Melting aluminum chips, Castings, vol. 38, 1910, p. 78.

The results of the experiments with the five fluxes named follow:

Results of melting borings with fluxes containing fluorides.

Run No.	Chips from barrel—	Observer.	Charge.	Flux used.	Amount of flux, on basis of weight of chips.	Approximate final temperature.	Yield of metal.		
							Primary yield, based on weight of chips.	Total yield, based on weight of chips.	Total yield, based on metal content of chips.
			Kg.		*Per cent.*	° *C.*	*Per cent.*	*Per cent.*	*Per cent.*
41	A	G	1	No. 1	25	900	71	72	80
42	A	G	1	No. 2	25	900	64	71	79
43	A	G	1	No. 3	25	900	65	69.5	78
44	A	G	1	No. 4	1.5	900	54	65	72
45	A	G	1	No. 4	20	900	50	62	70
46	A	J	1	No. 5	15	900	68.5	71	79
47	A	G	1	No. 5	25	900	70	71.5	79.5

In runs Nos. 44 and 45, with the soda ash, pearl ash, borax, cryolite flux, which is low in fluorine, the dross showed a thermit-like reaction, whereas the other fluxes gave a pasty mixture of dross and flux which cooled without such reaction. The four fluxes, Nos. 1, 2, 3, and 5, gave consistent results, better than the average results from any other of the fluxes used thus far.

The foundry reporting the results on tests with molten covers also stated that a small laboratory scale test in a crucible in a gas-fired furnace, in which millings free from oil or dirt, being cut without lubricant as a sample for analyses, and rather larger than commercial borings and free from very fine material, with a large proportion of powdered cryolite as flux, had given a recovery of 94½ per cent, although some of the metal was in the form of buttons, as the temperature was not high enough to make the cryolite sufficiently fluid.

TESTS WITH SALT AND FLUORSPAR FLUX.

As the writers learned at this point in the work that a flux of salt and fluorspar was being used commercially, especial attention was directed to that flux, on account of its cheapness, although its melting point is considerably above that of aluminum or its common alloys. As definite information on the proportions of the two ingredients used was not available, experiments were made on various mixtures of the commercial salt and fluorspar that were on hand. The freezing points of mixtures ranging from 90 parts NaCl and 10 parts CaF_2 (about 790° C.) to 75 parts NaCl and 25 parts CaF_2 (about 800° C.) were studied. The mixture of 85 parts NaCl and 15 parts CaF_2, which froze at about 785° C., had the lowest melting point and did not give so viscous a melt as those higher in CaF_2, being quite fluid at 800° C., was chosen as the most suitable composition. One mixture of 85 parts commercial $CaCl_2$ (containing NaCl) and 15 parts CaF_2

froze at about 660° C. On substituting 5 and 10 per cent of NaCl for similar percentages of $CaCl_2$, mixtures freezing at about 675° C. and 680° C. were obtained, but all of the $CaCl_2$ mixtures seemed very viscous at temperatures well above their fusion points and they did not seem to be better fitted for the purpose than the salt and fluorspar mixture, notwithstanding their lower freezing points, and they had the disadvantage of being hygroscopic.

In these runs the flux was intimately mixed with the chips before charging. The borings were rammed down as soon as they got soft and pasty so as to make room for more borings. After the whole charge was in, the crucible was not disturbed or stirred until the temperature of the flux on top of the metal was 900° C., at which temperature it is fluid enough, unless there is too much dirt present in proportion to the amount of flux used, when higher temperatures are needed to make the mass really fluid. Then the crucible was taken from the furnace, stirred gently—too violent stirring will carry globules of metal up into the slag from the metal below—skimmed, and poured. The buttons were recovered from the slag as in the previous experiments.

Results of melting borings with flux consisting of 85 per cent NaCl and 15 per cent CaF_2.

Run No.	Chips from barrel—	Observer.	Charge used.	Quantity of flux, based on weight of chips.	Approximate final temperature.	Yield of metal.		
						Primary yield, based on weight of chips.	Total yield, based on weight of chips.	Total yield, based on metal content of charge.
			Kg.	*Per cent.*	*°C.*	*Per cent.*	*Per cent.*	*Per cent.*
48	A	J	1	15	900	57.5	64	71
49	A	J	2	25	900	68.5	70	78
50	A	J	2	25	900	74	75	83.5
51	A	J	1	25	900	69	72	80
52	A	G	1.5	50	900	70	76	84.5
53	A	J	1	50	900	63	68	75.5
54	A	J	1	100	900	53	63.5	70.5
55	A	J	1.5	20	900	61	65	72

In run 55 the borings and flux were puddled, as in melting with $ZnCl_2$ in the iron-pot method, and kept pasty by continued stirring till all the charge was in, then at the end the temperature was raised. Apparently nothing is to be gained by so doing, as compared with merely ramming the borings down to make room for the whole charge, and not stirring till the end.

The behavior with only 15 per cent flux or with as much as 100 per cent was analagous to that with similar amounts of salt alone. About 25 to 50 per cent of this flux appears advisable for borings carrying the amount of dirt contained in the lot melted.

SUMMARY OF RESULTS WITH VARIOUS METHODS OF MELTING.

A summary of the results of some of the experimental runs on this consignment of dirty chips, by the chief methods that have been suggested, is shown below:

Results of melting borings by various methods.

No. of run.	Flux or method used.	Number of tests.	Average total recovery, on basis of metal content of charge.	Maximum.
			Per cent.	*Per cent.*
1	No flux used	1	54.5	....
2 to 7	NH_4Cl flux used	6	69.5	77
8 to 10	$ZnCl_2$ flux used	3	66	74
11	$AlCl_3$ flux used	1	72	....
14, 15	Puddling, with NH_4Cl added at end of melt	2	74.5	78
16	Retorting, with NH_4Cl added at end of melt	1	71.5	....
26 to 30	Various molten covers, other than NaCl used	5	66	73.5
32 to 39	NaCl in proportions of 15 to 100 per cent	8	74	85
33 to 38	Common salt in proportions of 20 to 50 per cent used	6	75	85
40	$NaCl-CaCl_2$ flux used	1	80	....
41 to 47	Various fluoride fluxes other than $NaCl-CaF_2$ used	7	79.5	80
48 to 55	$NaCl-CaF_2$ flux used	8	75	84.5
49 to 53, 55	$NaCl-CaF_2$ flux in proportions of 20 to 50 per cent used	5	80	....

The best results appear to come from the three following methods: Using NaCl, NaCl plus $CaCl_2$ in rather large proportion, or from the fluoride-containing fluxes in rather large proportion. Of the fluoride-containing fluxes the cheapest, the $NaCl-CaF_2$ flux, is apparently as good as those more expensive and seems to give, on an average, sufficiently better results than NaCl without CaF_2 to justify the use of the CaF_2.

In the type of furnace used for the tests, which was not properly adapted to puddling, the use of salt and fluorspar without puddling gave better results than puddling and with much less labor.

This flux was then tested out experimentally under other conditions on briquetted material in a small reverberatory furnace and in an electric furnace.

EXPERIMENTS WITH BRIQUETTED CHIPS.

As is brought out in page 19, compacting the borings into briquets under heavy pressure has been suggested on the theory that such briquets could be more readily plunged under a heel of molten metal than the loose borings, and that the briquetted metal would act much like ingot metal.

However, one refining plant, melting in coke-fired pit furnaces and using sal ammoniac as flux, had some borings briquetted. The briquets were very solid and compact, and could be thrown about without breaking. No quantitative data are available on the results obtained with them; but it was reported that they disintegrated on melting, and, save for greater ease in charging and possibly greater speed in melting, their use had no advantage over charging loose borings, as the loss was about the same.

A firm selling briquetting machines sent the writers two briquets, stating that they were made from a class of shop fines of No. 12 aluminum containing, unavoidably, a considerable amount of dirt, and that "this class of briquets refused to melt without unreasonable loss." These briquets, which were approximately disks or flat cylinders 3 inches in diameter, 1 inch thick at the edge and 1½ inches thick at the center, were very solid and had apparently been made under extremely high pressure. The briquets were run down with results as follows:

RESULTS OF MELTING BRIQUET NO. 1.

In run No. 56 a heel of metal, consisting of 693 grams of clean No. 12 ingot, was melted in a No. 5 crucible in a gas furnace and raised to a temperature of 800° C., when one of the sample briquets, weighing 342 grams, was put in. It sank in the molten metal, a smoky flame, evidently caused by the cutting oil used in machining the castings from which the chips came, being given off. The briquet was poked from time to time with a graphite rod. It disintegrated to some extent and much light dross rose to the surface. Seven minutes after charging the bath was again at a temperature of 800° C., and 7 grams of anhydrous zinc chloride was added and stirred into the dross. The dross showed a tendency to glow and heat up, similar to the thermit reaction, indicating that there were still very tiny globules of metal in the dross. The dross was skimmed off into water and the metal poured into ingot. Although the metal had been well stirred, much dross stuck to the bottom and sides of the crucible. After pouring the metal this dross was also skimmed into water. The dross was then washed on a 14-mesh sieve and all buttons over 14 mesh were collected.

In all, 920 grams of ingot were poured and 7 grams of buttons were collected. Deducting the 693 grams of heel metal, there was recovered from the original 342-gram briquet 227 grams of ingot and 7 grams of buttons, or the total recovery was 234 grams. On an assumed basis of 90 per cent metal content and 10 per cent oil and dirt in the briquet, the results are as follows:

Results of melting briquet in heel of molten metal (run No. 56).

	Per cent.
Primary yield, on basis of weight of briquet	66.5
Secondary yield, on basis of weight of briquet	2.0
Total recovery, on basis of weight of briquet	68.5
Total recovery, on assumed basis of 90 per cent metal content	76.2

This recovery is not much above the results from loose borings of apparently the same quality as those in the briquet when melted by this method.

RESULTS OF MELTING BRIQUET NO. 2.

Briquet No. 2 was melted with a flux consisting of 85 per cent common salt and 15 per cent fluorspar. As the flux could not be intimately mixed with the briquet, as can be done with loose borings, it was decided to cover the briquet with the flux. In this run (No. 57) it was impossible to cover the briquet in a crucible with so little as 20 to 30 per cent of its weight of this flux, the amount required for loose borings. Accordingly, 200 grams of flux was melted in a No. 5 crucible, and when it had reached a temperature of 800° C. the briquet, weighing 345 grams, was charged. As the flux did not quite cover the briquet, 100 grams more flux was added on top. In 12 minutes the briquet and the added flux had melted and the whole was at 875° C. The flux was then poured off, the metal poured into ingot, and the pasty mass of dirt which had soaked up flux and stuck to the sides of the crucible on pouring was scraped out. The flux and scrapings were disintegrated in water, after being permitted to cool somewhat, and washed on a 14-mesh sieve. In all, 265 grams ingot and 15 grams buttons were recovered, a total of 280 grams. Or the recovery was as follows:

Results of submerging briquet in molten flux (run No. 57).

	Per cent.
Primary yield, on basis of weight of briquet	76.8
Secondary yield, on basis of weight of briquet	4.2
Total recovery, on basis of weight of briquet	81.0
Total recovery, on assumed basis of 90 per cent metal content	90.0

CONCLUSIONS AS TO TESTS.

This recovery is better than that obtained on the loose dirty borings of lot A, but the briquets, although they contained some oil and dirt, did not appear to contain quite so much as lot A, hence no conclusions can be drawn from this single small-scale test as to any decrease in melting loss due to briquetting.

Briquetting will increase the ease of handling and probably increase the speed of melting, as the solid briquets transmit heat more rapidly than loose material. Whether it would increase the recovery or not, and whether the cost of briquetting, including labor, power, interest, and upkeep on an expensive press, would be compensated for by increased recovery and decreased labor and fuel cost in melting, can only be determined by large-scale tests on loose and briquetted chips from the same consignment.

In order to utilize the salt-fluorspar flux, it might be worth while to mix 20 to 30 per cent of flux, depending on the amount of dirt in the borings, with dirty borings before briquetting, thus making a briquet with the flux just where it is needed.

REVERBERATORY MELTING.

One firm is refining aluminum borings in a reverberatory furnace and claims 80 per cent recovery. The details are not available as the refiners claim to have a "secret process." Reverberatory melting is also done to a small extent by the firm using the $NaCl-CaF_2$ flux in crucible melting. This flux is also used when they melt in the reverberatory. Reverberatory melting of aluminum borings is considered by many refiners as out of the question, because of loss by oxidation in melting; also most of the firms melting aluminum ingot consider reverberatory or open-flame melting impossible for the same reason. On the other hand ingot may be successfully melted in an open-flame furnace if a strongly reducing flame is used, and the market alloys put out by the manufacturers of aluminum are usually made in large reverberatory or tilting open-flame, natural gas, or oil furnaces.

In order to determine whether the salt-fluorspar flux, which in crucible melting has consistently given better results than any other flux or method used in the experimental tests, was applicable to reverberatory melting, a small make-shift reverberatory or tilting open-flame gas furnace was built, an old carboy rocker being used for the tilting device, and an old sink for the body of the furnace.

The sink was lined with fire brick and a hearth laid within the fire-brick shell and lined with alundum-magnesite mixture. The hearth proper was about 2 inches deep at the lowest point, 2½ inches wide, 10 inches long on the flat part of the bottom, and sloped up to 5 inches wide by 16 inches long at the top of the hearth. A pouring spout was made at one end and space for a charging hole left above the hearth at the other. An arched fire-brick roof was added, the highest point of which was about 5 inches from the flat bottom of the hearth, or 3 inches above the level of a full molten charge, and 1 inch above the top of the hearth proper at the edges of the hearth on the sides of the furnace. The walls and roof were all 4 inches thick.

City gas, the only kind available, was used to heat the furnace. A home-made burner made from pipe and fittings was placed a few inches in front of the pouring spout. The waste gases passed out the charging hole of the furnace. As the burner was not tightly luted into the mouth of the furnace, but the flame was merely directed into it, air was freely drawn in by injector action. The flame did not heat the furnace very uniformly, the hottest part being about one-third the way from the front. The rear third of the furnace did not heat up very well, so the furnace was run by tilting it forward a little and utilizing only the front half, the rear being used to preheat the charge, which was poked forward as room was made for it at the front.

It will be seen that it would be difficult to maintain a very strongly reducing flame in such a furnace, so freely open to the air. The temperature varied so throughout the furnace that except on charges melted to a uniform liquid, the temperature of the charge varied with the location in the furnace.

A number of melts of aluminum borings from lot B, containing oil and much fine dirt, with not over 85 per cent metal content, were made. The results of these melts are given below in the order in which they were made:

TESTS IN REVERBERATORY FURNACE.

Run No. 58.—Four pounds of borings were mixed with 1 pound (85 per cent NaCl, 15 per cent CaF_2) of flux and the whole charged into the furnace, which was fairly hot at the start. The run lasted 22 minutes, no attempt being made to keep the flame strongly reducing. The metal was poured before the flux was really fluid, the temperature in the hottest part of the flux being 825° C. Apparently the amount of flux was too small, and the charge a little too large, as the direct flame played on the upper part of the charge. Some thermit-like burning of the charge near the front of furnace was noted.

In this run 1.6 pounds of ingot and 2 pounds of dirty buttons over 20-mesh in size, mixed with balled-up masses of dirt and flux, were recovered. The buttons were remelted with 1 pound of flux, yielding 1.05 pounds ingot, and 0.2 pound of large buttons, the total recovery being 2.85 pounds. The primary yield was 40 per cent, the secondary yield 31 per cent, making a total of 71 per cent, or 83½ per cent on the basis of metal content of charge.

Run No. 59.—The whole charge, consisting of 3.2 pounds borings and 1 pound of NaCl–CaF_2 flux mixed with borings, plus 1 pound flux spread on top after charging, was charged at the start in a hot furnace. The furnace was run 25 minutes and the bulk of the metal poured, then run 15 minutes more to melt the flux, but it was pasty at the end of the run. No thermit reaction was noted. The yield was as follows: Primary yield, 2.4 pounds, 75 per cent; 0.05 pound large buttons, 1.5 per cent; total, 2.45 pounds, 76.5 per cent, or 90 per cent on the basis of metal content of charge.

Run No. 60.—The whole charge, consisting of 3 pounds of borings with 1 pound of flux spread on top, none mixed in with borings, was charged in the furnace, which was warm, but not hot. The run lasted 25 minutes. At times thermit-like burning of the charge was observed. The dross and flux were very dry, not being fluid or even pasty, though the temperature in the hottest part of the furnace was over 900° C.

The recovery was as follows: Primary yield, 1.55 pounds, 52 per cent; 0.2 pound of large buttons, or 6½ per cent; total yield, 1.75 pounds, or 58½ per cent, or 69 per cent on the basis of metal content of charge.

Run No. 61.—Three pounds of borings and 1 pound flux mixed were charged in the hot furnace and ½ pound of flux spread on top. The run lasted 25 minutes; the flux was pasty at the end. No thermit action was observed. The recovery was as follows: Primary, 2.1 pounds, or 70 per cent; large buttons, 0.05 pound, or 1½ per cent; total, 2.15 pounds, or 71½ per cent, or 84½ per cent on the basis of metal content of charge.

Run No. 62.—The whole charge, consisting of 3 pounds of borings and 2 pounds flux, all mixed in, was charged in the hot furnace; ran 20 minutes. The flux was nicely pasty and a little over 900° C. in the hottest part. No thermit action was noted. The recovery was as follows: Ingot, 2.1 pounds, or 70 per cent; large buttons, 0.05 pound, or 1.5 per cent; total, 2.15 pounds, or 71½ per cent, or 81.5 per cent on the basis of metal content of charge.

Run No. 63.—The whole charge, consisting of 5 pounds of borings and 2½ pounds of flux, all mixed in, was charged in the hot furnace; ran 30 minutes. The charge was too large, as the flame played on top of it. Some thermit action was noted. The mixture of flux and dross was too dry.

The recovery was as follows: Ingot, 2.9 pounds, or 58 per cent; large buttons, 0.15 pound, or 3 per cent; total yield, 3.05 pounds, or 61 per cent, or 71.5 per cent on the basis of metal content of charge.

Run No. 64.—The whole charge, consisting of 3 pounds of borings and 3 pounds of flux, all mixed in, was charged in the hot furnace; ran 20 minutes. The flux was fully fluid at the end of the run.

The recovery was as follows: Ingot, 2.1 pounds, or 70 per cent; large buttons, 0.25 pound, or 8 per cent; total yield, 2.35 pounds, or 78 per cent, or 92 per cent on the basis of metal content of charge.

Run No. 65.—Three pounds of borings, mixed with 1.5 pounds of flux, was charged in the hot furnace and 1 pound of flux spread on top; ran 20 minutes. The flux was nicely pasty.

The recovery was as follows: Primary yield, 2.2 pounds, or 73.5 per cent; large buttons, 0.05 pound, or 1½ per cent; total yield, 2.25 pounds, 75 per cent, or 88 per cent on the basis of metal content of charge.

Run No. 66.—Three pounds of borings were used. No flux was added till the end, then 0.15 pound of sal ammoniac was added and stirred in; the run lasting 17 minutes. The flame used was the same as in previous runs with other fluxes. The melt was only stirred occasionally; very decided glowing or thermit-like action was obtained. The dross was skimmed out on an iron plate and dumped into water at short intervals during skimming.

In this run 1.15 pounds ingot was obtained and many small buttons mixed with adhering dross, which were remelted and yielded 0.1 pound of ingot, 0.05 pound of large buttons; the total yield being 1.3 pounds. The primary yield was 38.5 per cent; the secondary yield, 5 per cent; and the total, 43.5 per cent, or 51 per cent on the basis of metal content of charge.

Run No. 67.—Three pounds of borings were used, and no flux was added until the end of the melt, when 0.15 pound of $ZnCl_2$ was added and stirred in. The flame was on 12 minutes, the charge being stirred all the time and the borings worked over with a skimmer. The flame was kept as strongly reducing as possible, almost smoky, being much less oxidizing than in previous runs. Not much thermit action was observed until the borings were nearly all melted down, when the whole mass started to heat up almost instantly. The flame was cut off and the mass vigorously stirred for 3 minutes more, then $ZnCl_2$ was added. The dross was skimmed off as in run 66.

The recovery was as follows: Primary yield, 1.1 pounds, or 37 per cent; large buttons, 0.35 pound, or 11.5 per cent; total yield 1.45 pounds, or 48½ per cent, or 57 per cent on the basis of metal content of charge.

Run No. 68.—The whole charge, consisting of 3 pounds of borings with three-eighth pound $NaCl-CaF_2$ flux mixed in, and three-eighth pound spread on top was charged in the hot furnace; ran 16 minutes. The flame was not smoky. The flux and dross were too dry.

The recovery was as follows: Ingot, 1.6 pounds, or 53½ per cent; large buttons, 0.25 pound, or 8½ per cent; total, 1.85 pounds, or 62 per cent, or 73 per cent on the basis of metal content of charge.

When the runs are tabulated in the order of the amount of NaCl–CaF_2 flux used, the results may be summarized as follows:

Summary of results in tests with reverberatory furnace.

Run No.	Flux used.	Quantity of flux, based on weight of chips.	Method of adding flux.	Yield of metal—			
				On basis of weight of chips.			Total yield on basis of metal content of charge.
				Primary.	Secondary.	Total.	
		Per cent.		*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
66....	NH_4Cl..	5	At end of melt....................	38.5	5	43.5	51
67....	$ZnCl_2$..	5	.....do[a]	37	11.5	48.5	57
68....	85 parts NaCl, 15 parts CaF_2.	25	Half mixed in charge, half placed on top.	53.5	8.5	62	73
58....	...do....	[b]25	All mixed in charge[c]............	40	31	71	83.5
60....	...do....	33	All placed on top................	52	6.5	58.5	69
61....	...do....	50	Two-thirds mixed in charge, one-third placed on top.	70	1.5	71.5	84.5
63....	...do....	50	All mixed in charge[d]............	58	3	61	71.5
59....	...do....	60	One-half mixed in charge, one-half placed on top.	75	1.5	76.5	90
62....	...do....	66	All mixed in charge....	70	1.5	71.5	84.5
65....	...do....	83	Three-fifths mixed in charge, two-fifths placed on top.	73.5	1.5	75	88
64....	...do....	100	All mixed in charge..............	70	8	78	92

[a] Reducing flame, charge stirred constantly.
[b] 37 per cent including flux used on remelt.
[c] 4 pounds borings, not heated hot enough, 50 per cent of flux used on remelt of secondary yield.
[d] 5 pounds borings, charge too large, causing flame to strike it.

REMARKS ON RESULTS OF TESTS.

In run 68 evidently not enough flux was used, as the flux plus dross was dry, and the primary yield was low; that is, there was poor separation.

Run 58 showed even poorer separation, as the temperature was not raised high enough. Increasing the proportion of flux on the remelt made up for this somewhat.

Run 60 also shows a poor primary yield, the flux being all on top and not properly protecting the borings.

In run 63 the charge was too large, so that the flame played directly on much of the surface of the charge.

On account of the greater surface in the reverberatory than in the crucible, it is natural that a greater proportion of flux be required.

Runs 59, 61, 62, 64, and 65, with enough flux, of which a sufficient amount was mixed in, averaged a primary yield of 70 per cent and a total yield of 74.5 per cent, based on the weight of chips, or 87.5 per cent on the basis of metal content, as compared with a primary yield of 40 per cent and a total yield of 50 per cent, or 60 per cent on the metallic basis when NH_4Cl or $ZnCl_2$ was used for flux. That is, the tests, though on too small a scale to be conclusive, indicate that with a suitable flux to coat the globules from the melting of the tiny borings, reverberatory melting may be feasible and give as good results as crucible melting, whereas without such a flux reverberatory melting is undoubtedly not as good as crucible melting.

These tests, like all the previous ones, have shown better experimental results on the same lot of borings when the NaCl – CaF_2 flux was used than by any other of the methods advocated.

Robinson[1] has recently obtained a patent on a process of recovering metals in which he melts salt in a reverberatory furnace, then from time to time adds the aluminum-bearing refuse, mixed with 10 per cent of its weight of cryolite, to dissolve aluminum oxide, preferably operating with constant puddling. As this patent appeared after the experiments herein described were completed, no tests were made of this method.

ELECTRIC MELTING.

A further test of the NaCl – CaF_2 flux was made on a rather larger scale with a crucible electric furnace of the Hoskins type. Runs 17 to 21 had not indicated any appreciable advantage from melting in the reducing atmosphere of this type of furnace, and the furnace was used merely because it was the most convenient one at hand in the laboratory which would take larger charges than the small gas furnace.

EXPERIMENTS IN MELTING INGOT.

In three runs on ingot, in which a total quantity of 69 pounds of clean No. 12 ingot was remelted in this furnace without flux, the clean ingot poured weighed 68.5 pounds, also 0.5 pound of skimmings and spillings were obtained, of such metal content that at least 0.3 pound of clean metal would have been obtained on remelting them. That is, the actual metal loss was not over the one-third of 1 per cent given by good melting practice with the iron-pot furnace.

EXPERIMENTS IN MELTING BORINGS AND SKIMMINGS.

Three preliminary runs A, B, and C, were made on some odd lots of borings that had accumulated from the previous runs, hereinafter reported, on sizing the borings, and on some skimmings and spillings.

Run A was made on 16.65 pounds of miscellaneous borings, different from but approximately of the same quality as those used in run B, with which 20 per cent by weight of the flux consisting of 85 per cent NaCl and 15 per cent CaF_2 was mixed. The recovery, based on total weight of chips taken, was 11.8 pounds ingot, 0.6 pound buttons, or a primary recovery of 71 per cent and a total recovery of 74.5 per cent.

In run B, on 15 pounds of miscellaneous borings plus 20 per cent of the flux, the primary yield was 11 pounds ingot, or 73.5 per cent, and the total yield 12.2 pounds, or 81.5 per cent, on the basis of total weight of chips.

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[1] Robinson, C. S., U. S. Patent 1180435, April 25, 1916.

In run C, on 13.5 pounds skimmings, plus 20 per cent flux, recovery was 11 pounds ingot, or 11.25 pounds in all, or a primary yield of 82 per cent and a total yield of 83.5 per cent. Two runs were then made on chips from lot B with the following results:

Results of melting miscellaneous borings with NH_4Cl and $NaCl$-CaF_2 flux.

	Run No. 69.	Run No. 70.
Observers	G, L	G, L
Weight of chips taken, pounds	15	15
Flux used	NH_4Cl	85 parts NaCl. 15 parts CaF_2.
Percentage of flux used, on basis of weight of chips	2	20
Yield of metal:		
Primary yield, percentage of weight of chips	53	68.5
Total yield, percentage of weight of chips	58	71.5
Total yield, percentage of metal content of charge	68.5	84

As the crucible was loose in the furnace, not being supported at the sides, puddling was not feasible, so in run 69 the borings were poked and stirred quite constantly as long as oil was smoking off, but the metal was allowed to become more fluid between chargings than was the case in the puddling experiments.

In run 70 no stirring was done till the end, when the temperature was raised well above 900° C., so that the flux was really fluid.

COMMERCIAL TEST OF SALT AND FLUORSPAR FLUX.

As the iron pot, puddling process and the crucible, salt and fluorspar process appeared to be, from the experimental results and from the reports of the users of the different processes, the two most satisfactory in commercial use, it was suggested to the firm using the puddling process that to test that process against the salt and fluorspar process on the same lot of borings would be of interest. It was pointed out that the higher temperature required in the latter process to make the flux fluid enough to allow a good separation might be too high to permit practical life of the iron pot.

This firm made such a test, using an iron-pot furnace for both methods, and reported as follows:

> A lot of borings that had been passed through a magnetic separator was carefully weighed, mixed, and divided into two parts. One part was melted down by puddling, about 2 per cent of zinc chloride being used as final flux; the other part was mixed with 25 per cent of a flux consisting of 85 per cent salt and 15 per cent fluorspar. The cost of flux per 100 pounds of chips was 10.4 cents for zinc chloride and 10.8 cents for the salt and fluorspar flux. The chips contained 13.3 per cent oil and 0.6 per cent free iron left after commercial magnetic separation. The dross from each melt was put in water, concentrated in a tumbling barrel, dried, and assayed, by running down in a small crucible, for its metal content.
>
> *Test 1.*—1,049 pounds of chips plus 20 pounds of zinc chloride yielded 778 pounds of ingot and 27 pounds of concentrated skimmings containing 74 per cent metal, or 20 pounds, a total of 798 pounds, giving a primary recovery of 74.16 per cent and a total recovery of 76.06 per cent.

Test 2.—508 pounds of chips mixed with 126 pounds of the flux consisting of 85 per cent salt and 15 per cent fluorspar yielded 376 pounds ingot and 22 pounds concentrated skimmings of 62.5 per cent metal content, or 14 pounds, a total of 390 pounds, giving a primary recovery of 74.01 per cent and a total recovery of 76.71 per cent.

Test 1 took 6¼ hours and test 2 took 8 hours, or the ingot produced per hour was 119 pounds with the puddling method as compared with 47 pounds by the salt and fluorspar method; that is, the production was cut down 60 per cent. The iron pot used in test 2 was practically ruined at the end of the test, owing to the high temperature necessary to melt the flux. The metal is heated too hot during the melting, thus injuring the quality, in the salt and fluorspar method. The results show that the salt and fluorspar flux can not be used with an iron pot, for the flux has little effect if not pasty or molten, and the temperature necessary for this result is too high for the iron pot. The best place to use such a flux is in melting grindings or very dirty borings. When work slackens somewhat we are going to make a test with a crucible, and will inform you of the results.

No report has yet (June, 1916) been received on the crucible test. From some other comments it seemed possible that this firm had used continuous puddling on test 2 as well as on test 1, although in suggesting the test it was pointed out that continuous stirring was apparently not desirable with the salt and fluorspar flux. An inquiry on this point, and as to whether the borings contained dirt as well as oil was unanswered. From the comment on the apparent applicability of the salt and fluorspar flux to dirty borings, it would seem probable that the borings tested were not dirty.

If the true metal content be taken as the oil-free content, the recovery of 76 to 77 per cent obtained by both methods is equivalent to 88 to 89 per cent on the basis of true metal content, or well above the 68 to 70 per cent recovery, on the basis of original weight of chips, reported by this firm as its average yearly recovery on all borings melted by the puddling process, which would indicate that the borings used were of better quality than the average.

Although the results of this test are unfavorable to the use of salt and fluorspar flux for iron-pot melting, they do not show how puddling in an iron pot and using the salt and fluorspar flux in coke or oil-fired crucible furnaces would compare. The output per furnace was less with the salt and fluorspar flux, but it should be remembered that when salt and fluorspar flux is used the charge does not require continuous stirring, as in the puddling process, and that the output per furnace tender should therefore not be reduced. Of course, the melting of 25 per cent of flux will require more fuel than in a process which does not use a large proportion of flux. Moreover, if the results of the small-scale tests on reverberatory melting would hold on a commercial scale, it would appear that the fuel and labor economies possible from melting in a reverberatory on a larger scale than with crucible or pot furnaces, might perhaps be better gained by using the salt and fluorspar flux, as puddling in a reverberatory would be very difficult.

EFFECT OF SIZING THE BORINGS.

In order to determine whether sifting the borings and melting the larger and the smaller sizes separately would be of advantage, some borings from lot A were sifted on a 20-mesh riddle, 80 per cent remaining on it and 20 per cent passing through it. The results of runs made on these borings were as follows:

Results of melting sized borings from lot A with NH_4Cl flux.

	Run No. 71.	Run No. 72.
Size of borings	Over 20-mesh.	Under 20-mesh.
Observer	G	G
Weight of chips used, kilograms	2	2
Percentage of flux, on basis of weight of borings	2	2
Yield of metal, on basis of weight of chips:		
Primary yield, per cent	50.5	8.4
Total yield, per cent	67.5	18.3

As the total yield on the larger borings, which make up 80 per cent by weight of the unsifted borings, was 67.5 per cent and that on the smaller borings, which make up 20 per cent of that weight, was 18.3 per cent, the total yield on the basis of weight of unsifted chips was $\frac{(67.5 \times 80) + (18.3 \times 20)}{100} = 58$ per cent, which is equivalent to 64.5 per cent of the actual metal content.

A 14-mesh riddle was used in order to get a better separation of larger borings from the smaller ones, and the material passing 14-mesh was sifted on a 40-mesh screen to free it from dirt, the material under 40-mesh being discarded. The two sizes were each melted with the NaCl–CaF_2 flux by heating to 900° C. The following results were obtained:

Results of melting sized borings from lot A with NaCl–CaF_2 and $ZnCl_2$.

Run No.	Lot.	Weight of charge.	Size of borings.	Observer.	Flux used.	Quantity of flux used, on basis of weight of chips.	Yield of metal, on basis of weight of chips.	
							Primary.	Total.
		Kg.				*Per cent.*	*Per cent.*	*Per cent.*
73	A	1.25	Over 14-mesh	J	85 parts NaCl, 15 parts CaF_2.	15	84	84.5
74	A	1.25	do	J	do	15	87	88
75	A	1.25	do	J	do	25	87	88
76	A	1.25	do	J	do	25	86	86.5
77	A	1	Between 14 and 40 mesh.	J	do	25	71	72
78	A	1	do	J	do	25	73.5	74
80	A	1	do	J	do	50	68	68.5
81	A	1	do	J	do	50	70.5	71
82[a]	A	1.5	do	J	$ZnCl_2$	2	56	61.5

[a] Run 82 was made by the puddling process.

In the runs in which the NaCl–CaF_2 flux was used the average total recovery on the material over 14-mesh was 87 per cent and on that between 14 and 40 mesh it was 71.5 per cent. As 54 per cent of the unsifted borings passed the 14-mesh riddle, 42 per cent was between 14 and 40 mesh, and 4 per cent was under 40-mesh and discarded, being practically all dirt. The results may be figured as follows: $\frac{(54\times87)+(42\times71.5)}{100}=77$ per cent, which is the total recovery on the basis of the original weight of dirty borings, or 85.5 per cent on the basis of metal content, a recovery that is only slightly better than the average of results on unsifted material and is the same as the maximum. On the very fine material the use of salt and fluorspar flux gave markedly better results than puddling, which suggests that the borings might be sifted and the puddling process used on the cleaner and larger fraction, thus allowing the use of the iron pot for most of the chips, and the smaller and dirtier part melted in crucibles in coke or oil fires with NaCl–CaF_2 flux. This method would reduce the fuel consumption, as more would be required to melt the large amount of NaCl–CaF_2 flux needed were it to be used on all of the borings without sifting them.

Some chips from lot B were also sifted on 14 and 40 mesh riddles, giving 57.5 per cent over 14-mesh, 30.3 per cent between 14 and 40 mesh, and 12.2 per cent under 40-mesh, which was practically all dirt, and discarded. A run (No. 83) was made on the material that was over 14-mesh, in the electric furnace used for runs 69 and 70. The results were as follows:

Result of melting chips from barrel B, over 14-mesh in size, in an electric furnace (run No. 83.)

Observers, G and L.
Weight of chips used, 15 pounds.
Flux used, 85 per cent NaCl and 15 per cent CaF_2.
Quantity of flux used, based on weight of chips, 20 per cent.
Yield of metal, on basis of weight of chips:
    Primary yield, 86.5 per cent.
    Total yield, 89 per cent.

This larger scale test on the material over 14-mesh from lot B agreed well with the tests in the small gas furnace (runs 73 to 76) of the material over 14-mesh from lot A. The material from 14 to 40 mesh in size was used in washing tests.

As the large chips are much easier to handle so as to get a high recovery than those under 20-mesh, it would pay to separate them for remelting if some use could be found for the fine material. Where, as in the case cited on page 10, there is a demand for chips for chemical use, this could be done. Possibly the fine material might be used in calorizing, after further comminution, and would offer a cheaper source of aluminum for that purpose than the aluminum powder now used, provided the copper or zinc in the alloy chips was not detrimental, and provided also that the dirt in the chips would take the place of the aluminum oxide now added to prevent coalescence.

EXPERIMENTS IN WASHING THE CHIPS.

The results in all of the experiments described go to show that clean borings may be melted with comparatively little loss, and that a small content of fine dirt greatly increases the difficulty of making the tiny globules coalesce. When the borings are oily, the dirt sticks tightly to them, so that simply sifting them will not remove the dirt.

Obviously the next experiment to try, then, was to wash the borings so as to free them from oil and then sift out the dirt.

WASHING WITH HOT WATER.

An attempt was made to wash chips from lot A with hot water, in the hope that the oil would be freed from the chips and float to the top. But although the chips in lot A contained about 3 per cent oil, as was determined by ether extraction, not a trace of oil was floated off by the water. Bancroft[a] has explained this retention of oil by the fact that aluminum adsorbs oil more readily than it does water. He has found in flotation experiments that if aluminum powder be shaken with kerosene and water, the aluminum goes entirely into the kerosene layer, being floated up by it. In the experiment by the writer the chips, being too heavy to be floated by the small amount of adsorbed oil, held the oil down with them instead of rising with it as in the case of the powder in the presence of much oil.

WASHING WITH A WATER-SOLUBLE CUTTING COMPOUND.

Inasmuch as a high recovery was reported on chips cut with a water-soluble cutting compound and then centrifuged, it was thought possible that oily borings washed with this compound might be cleaned and freed from dirt, so some of the compound was obtained through the courtesy of the makers.

This compound was a stiff white emulsion of oil, soda soap, and water, the water forming over half of the material. On ignition it formed 2 to 2.5 per cent ash, mainly Na_2CO_3. It was practically neutral in reaction, showing a very faint alkaline reaction with phenolphthalein, due to hydrolysis of the soap. When the compound was stirred into water a very stable milk-white emulsion was formed.

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[a] Bancroft, W. D., In discussion. Trans. Am. Electrochem. Soc., vol. 23, 1913, p. 294.

Washing chips from lot A with this emulsion did not remove appreciable amounts of oil or dirt, so that the high recovery reported seems due to cleanliness rather than to any specific action of the cutting compound.

WASHING WITH GASOLINE.

Washing with gasoline was then attempted. The borings were washed on a 20-mesh riddle and the fraction over 20-mesh and that under 20-mesh were dried separately. The dry material under 20-mesh was then sifted on an 80-mesh riddle. Only one change of gasoline was used, but it took nearly 5 gallons of gasoline to wash about 60 pounds of borings. The results of washing were as follows: Oil eliminated, 3 per cent; dirt eliminated, 7 per cent; clean borings over 20-mesh, 72 per cent; clean borings under 20-mesh, 18 per cent.

The borings over 20-mesh were quite bright, but there was still some oil not yet washed out and some dirt adhering to them, and on melting down these borings oil still smoked off, though the smoking stopped much sooner than with the original unwashed material. The washed borings under 20-mesh contained a little more oil than those over 20-mesh, and considerably more dirt. As no accurate method of analysis to determine the amount of dirt still retained could be devised, the assumption was made for purposes of computation that the borings after washing were 100 per cent metal.

A heat of washed borings was then run with a mixture of 72 parts of borings over 20-mesh and 18 parts of borings under 20-mesh—that is, a mixture that could be considered as washed but not sifted. The results of this run were as follows:

Results of melting borings washed in gasoline (run No. 83).

Chips from lot A used in test.
Observer, G.
Weight of chips used, 2 kilograms.
Flux used, NH_4Cl.
Quantity of flux used, based on weight of chips, 2 per cent.
Yield of metal, on basis of weight of chips:
    Primary yield, 60 per cent.
    Total yield, 67 per cent.

As this charge weighed but 90 per cent of what the unwashed borings would weigh, and the washed borings are assumed to be all metal, the total yield given, 67 per cent, is on the basis of metal content, and the total yield on the basis of original weight of chips is 60 per cent.

Samples of the two fractions were then run separately, with results as follows:

Results of melting the fractions separately.

	Run No. 84.	Run No. 85.
Weight of chips used, kilograms	....	....
Observer	G.	G.
Size of borings	Over 20-mesh.	Under 20-mesh.
Flux used	NH_4Cl	NH_4Cl
Percentage of flux used on basis of weight of chips.	2	2
Yield of metal, on basis of weight of chips:		
Primary yield, per cent	73.5	26.8
Total yield, per cent	81.5	37.3

The average total yield, on the basis of the original weight of unsifted borings, is $\frac{(72\times 81.5)+(18\times 37.5)}{100}=65.5$ per cent, or on the basis of true metal content is 73 per cent. Samples of the two fractions were also run separately, poking each into a heel of 1 kilogram of molten metal, with the following results:

Results of melting borings with heel of molten metal.

Run No.	Chips used.	Charge used.	Size.	Observer.	Flux used.	Quantity of flux used on basis of weight of charge.	Yield of metal on basis of weight of charge.		
							Primary.	Total.	Average.
		Kg.				*Per cent.*	*Per cent.*	*Per ct.*	*Per ct.*
86	Chips from lot A, washed with gasoline.	1	Over 20-mesh.	G	NH_4Cl	2	70	80.5	....
87	do	1	Under 20-mesh.	G	do	2	29.5	41.5	40.5
88	do	1	do	G	do	2	23.5	39.5	

By this method the total yield, on the basis of weight of unsifted borings, is $\frac{(72\times 80.5)+(18\times 40.5)}{100}=65$ per cent, or on the basis of true metal content is 73 per cent.

When runs 84 to 88 are compared with runs 2 to 7 and 24, 25, in which similar melting methods were used on unwashed, unsifted material it appears that there is probably a slight advantage, as regards recovery, in washing and sifting.

Some of the material over 20-mesh washed with gasoline was melted with the $NaCl–CaF_2$ flux, with the following results.

Results of melting washed borings over 20 mesh in size with $NaCl-CaF_2$ flux.

	Run No. 89.	Run No. 90.
Observer	J.	J.
Size of borings	Over 20-mesh.	Under 20-mesh.
Weight of charge used, kilograms	1.25	1.25
Percentage of flux used, based on weight of borings	25	25
Yield of metal, on basis of weight of borings used:		
Primary yield, per cent	86.5	87
Total yield, per cent	87.5	88

The results check with those obtained on melting unwashed borings over 14-mesh with this flux in runs 73 to 76 on chips from lot A and in run 83 on chips from lot B, but are not superior, so that if washing is of advantage its advantage is mainly on the finer borings.

WASHING WITH CAUSTIC-SODA SOLUTION.

No runs were made with this flux on the fraction under 20-mesh, washed in gasoline, as the supply of this fine material was exhausted and gasoline washing seemed to be too expensive a process for commercial use. However, some of the material between 14 and 40 mesh left from the sizing of part of lot B for run 83 was washed in a dilute solution of caustic soda. Caustic soda or caustic potash in dilute solution, is used as a cleaner before polishing aluminum articles. If saponifiable oil or grease is present, it will form a soap that will tend to emulsify paraffin oil. If there is no saponifiable matter in the cutting oil, soap could be added to the NaOH or KOH solution.

Preliminary tests showed that the oil and dirt in chips of lot B were so loosened by the action of a 0.5 per cent solution of NaOH that if the chips were placed in the solution, allowed to remain with constant stirring till slight chemical attack of the NaOH on the aluminum had begun, as shown by a fairly rapid evolution of gas bubbles, a great deal of the dirt was brought to the top by the gas and could readily be floated off with water, which also serves to wash out the caustic solution.

After the chips had been washed, they were freed from water by centrifuging and then drying at 100° C.

The method of washing was rather crude, 1,500 grams of the chips between 14 and 40 mesh being placed in a pan, 5 liters of 0.5 per cent NaOH solution added, and the whole stirred till there was a fairly copious evolution of gas, which required about 10 minutes. Water was then run into the bottom of the pan through a tube and allowed to overflow with constant stirring of the chips until the water ran clear, which took about 10 minutes more.

The chips were then drained and centrifuged. The only centrifuge available was of very small capacity and there was a slight mechanical loss in transferring the chips. From 1,500 grams of the 14 to 40 mesh chips, 1,412 grams of slightly damp chips were obtained after centrifuging, which, after being dried at 100° C. and 15 grams of dirt sifted out on an 80-mesh screen, weighed 1,362 grams—that is, 9 per cent of the 1,500 grams of the 14 to 40 mesh fraction, or 3 per cent of the unsifted dirty borings, was removed from this fraction by washing. The 1,362 grams were then run down with 20 per cent of the NaCl–CaF_2 flux.

The results of the test were as follows:

Results of melting chips 14 to 40 mesh in size, washed in NaOH solution (run No. 89).

Chips used, from lot A, 14 to 40 mesh, washed in NaOH solution.
Observer, J.
Weight of chips used, 1.33 kilograms.
Flux used, 85 per cent NaCl, 15 per cent CaF_2.
Quantity of flux used, based on weight of charge, 20 per cent.
Yield of metal, on basis of weight of chips:
  Primary yield, 72 per cent.
  Total yield, 75.5 per cent.

On the basis of the 1,500 grams originally taken, the total recovery was 68.5 per cent, which is slightly less than the yield of 71.5 per cent obtained in runs 77 to 81, which were made on the unwashed 14 to 40 mesh fraction from lot A, but it must be remembered that the chips in lot A were not so dirty as those in lot B.

As the recovery on the unwashed fraction over 14 mesh in size used in run 83 was 89 per cent, the total recovery, calculated on the basis of original weight of unsifted material, is

$$\frac{(57.5\times 89)+(30.3\times 68.5)}{100}=72 \text{ per cent,}$$

or, on the basis of true metal content, 85 per cent.

Two 1,500-gram samples of the unsifted borings from lot B were then washed with NaOH solution, centrifuged, and dried, one yielding 1,326 grams and the other 1,316 grams. These samples were run down with 20 per cent of NaCl-CaF_2 flux, with results as follows:

Results of melting unsifted borings from lot B, washed in NaOH solution.

	Run No. 90.	Run No. 91.
Observer	J	J
Weight of borings used, kilograms	1.5	1.5
Flux used	80 parts NaCl 15 parts CaF_2	80 parts NaCl 15 parts CaF_2
Quantity of flux, on basis of weight of chips, per cent	20	20
Yield of metal:		
Primary yield, percentage of weight of chips	71	70
Total yield, percentage of weight of chips	74.5	75
Total yield, percentage of metal content of chips	87.5	88.5

These results show enough improvement over those on unwashed chips from lot B to make it seem possible that washing dirty borings with dilute NaOH solution might be worth while. One method that suggests itself for trial on dirty borings is to use a concentrating table of the Wilfley type, fed not with water but with dilute NaOH solution, or with a solution of both NaOH and soap, the solution to be run into a settler and pumped back into the system, followed by washing with water and centrifuging.

From the comparative readiness with which dirt was freed from the chips in the experiments it would seem that such treatment would be worth a commercial test. As most refining plants and many nonferrous foundries are now equipped with concentrating tables for use on brass-foundry wastes, the only unusual piece of apparatus required would be the centrifuge.

Borings thus cleaned and dried would probably be more readily separated from free iron on being passed over the magnetic separator than oily borings. Were a centrifuge installed, very oily borings, such as contain 10 per cent or more of oil, might be run through the centrifuge before washing, which would leave less work for the cleaning solution to do, and the cost of the operation would probably be compensated for by the value of the oil recovered.

MISCELLANEOUS EXPERIMENTS IN ELECTRIC FURNACES.

After the experiments above cited had been completed, another indirect arc furnace was constructed for other purposes, and the question arose whether or not the furnace would be applicable to melting aluminum chips.

Another lot of No. 12 alloy chips, designated as lot D, was obtained from a large refining company, which stated that it had made a 77 per cent recovery, on the basis of the weight of the chips, from these chips by the puddling process.

On chips that were sized without extracting the oil, the recoveries were as follows: Chips over 14 mesh in size, 62 per cent; between 14 and 40 mesh, 30 per cent; and under 40 mesh, 8 per cent. By extraction with ether, 5 per cent of oil was obtained; and by sifting the oil-free borings on a 40-mesh sieve, and carefully floating off the dirt from the small amount of aluminum that passed the 40-mesh sieve, 7 per cent of dirt was obtained. Hence the chips contained at least 12 per cent of nonmetallic material, and the metal content was not over 88 per cent. This means that in commercial practice the puddling process gave a recovery of 87.5 per cent of the metallic content.

EXPERIMENTS WITH INDIRECT ARC FURNACE.

The indirect arc furnace resembled in form a horizontal cylinder. The melting chamber was 15 inches in diameter and about 15 inches long. Graphite electrodes 2 inches in diameter were passed horizontally into the center of the chamber, and the arc was formed between them. The furnace lining forming the melting chamber was of fire brick, coated with asbestos cement, and this layer was coated with alundum magnesite mixture.

MELTING WITH ALUNDUM-MAGNESITE LINING.

Run No. 92.—Twenty-five pounds of chips, mixed with 10 pounds of flux, consisting of 85 parts NaCl and 15 parts CaF_2 was charged into the cold furnace. The charge came within about 2 inches of the arc. After 42 minutes heating in which 20 kilowatt-hours were used, a pool of metal had collected in the bottom, and the temperature was 900° C. The flux was not fluid and still held many buttons of metal, and a few unmelted chips remained in the bottom of the furnace. The yield of ingot was 6.25 pounds, or a primary yield of 25 per cent on the basis of original weight of chips charged. The flux was scraped out and on being crushed and washed yielded 8.25 pounds of buttons, or 35 per cent, which, with the 25 per cent previously obtained, made a total recovery of 60 per cent, on the basis of original weight of chips.

It was evident that the charge as a whole was not hot enough, and that it would have been better not to charge so many chips at once. The magnesite-alundum lining was badly attacked and began to crumble off, and as this was believed to be due to the effect of the fluorspar, it was decided to try common salt in the next heat.

Run No. 93.—Ten pounds of chips mixed with 5 pounds NaCl was charged into the cold furnace and the current turned on, 17 kilowatt-hours being used in 29 minutes, then 10 pounds of chips mixed with 5 pounds of NaCl was added. Heating was continued 25 minutes more, using 13 kilowatt-hours, or the total power input was 30 kilowatt-hours. The temperature was over 1,000° C, but the mass of NaCl and dross was only pasty. From the 20 pounds of chips charged, 7.5 pounds ingot and 5 pounds of buttons were obtained, or a primary yield of 37.5 per cent and a total yield of 62.5 per cent, on the basis of original weight of chips. The lining continued to crumble.

Run No. 94.—The furnace was preheated by the use of 15 kilowatt-hours and then 15 pounds of chips, plus 15 pounds of flux, consisting of 85 parts NaCl and 15 parts of CaF_2, was charged. Ten pounds of the flux was mixed with the chips, and 5 pounds placed on top of the charge. After 27 minutes, in which 15 kilowatt-hours more was used, the charge was at a temperature of 900° C. The bulk of the flux was fluid, but it contained some matted masses of flux and dross that were only pasty. Seven and one-half pounds of ingot and some flux were poured. The flux that poured out with the metal was replaced in the furnace and heated 20 minutes, in which 10 kilowatt-hours were used. Two and nine-tenths pounds ingot was obtained on the second pour. The flux was all fluid at this time. Besides the total of 10.4 pounds of ingot, 0.9 pound of buttons was recovered from the slag, giving a primary or ingot yield of 69.5 per cent and a total yield of 75 per cent on the basis of the weight of chips charged. The lining was still crumbling badly, and after the furnace had cooled all loose pieces were taken out. By this time most of the alundum magnesite layer of the lining had come off. It appeared that with a lining not attacked by the flux and with sufficient flux fair recoveries might be made.

Run No. 95.—In order to determine whether less flux than was used in the previous run would serve, run 95 was made, after the furnace had been preheated for 15 kilowatt-hours, by charging 18 pounds of borings mixed with 8 pounds of the flux (85 parts NaCl, 15 parts CaF_2) and placing 3 pounds of flux on top of the charge. After 44 minutes, in which 22.5 kilowatt-hours was used, there was a pool of metal in the furnace, and 10.2 pounds of ingot was poured, but the flux was not fluid. The heat was continued for 18 minutes, 7.5 kilowatt-hours being used, and 1.4 pounds more ingot was poured. The flux was still only pasty. Five-tenths pound of buttons was recovered from the slag, giving a combined primary or ingot yield of 64.5 per cent and a total yield of 67.5 per cent on the basis of chips charged.

The furnace was then cleaned, and a run made on a charge of 37 pounds of ingot. Starting with a cold furnace, it took 52 minutes' time and a power input of 30 kilowatt-hours to heat the charge to 735° C. Twenty-five pounds more of ingot was then added, and 10 more kilowatt-hours used in 13 minutes. The charge was then at 870° C., or hotter than is needed for pouring aluminum castings. Of course, after the furnace is once hot, the power needed to melt a given charge will be less than when the furnace must be heated also. Moreover, a furnace of commercial size will be much more efficient than a small laboratory furnace. Nevertheless, the power required for the electric melting of aluminum, even under the most favorable conditions, will be high. In this heat, which was started with a cold furnace, a total power input of 40 kilowatt-hours was required to heat 62 pounds of aluminum to 870° C., and 10 kilowatt-hours to melt 25 pounds and raise it to 870° C., and also to raise the first 37 pounds from 735° to 870° C. This furnace, started cold, has melted 125 pounds of red brass ingot and heated it to 1,150° C. with 40 kilowatt-hours, and after the furnace was hot 19 kilowatt-hours has melted and heated to 1,200° C. 130 pounds of red brass ingot.

That is, it took about twice as much power per pound to heat the aluminum to its pouring temperature as it did to heat red brass to its pouring temperature. This is in accord with theory. Richards[a] has calculated that theoretically about 278 heat units are required to heat aluminum to about 710° C., whereas only about 130 heat units are required to heat brass or bronze to about 1,050° C. The difference is due to the extremely high heat of fusion of aluminum, the relative heats of fusion being as follows: Aluminum, 100; copper, 43; zinc, 23; tin, 14; and lead, 4. This high heat of fusion overbalances the higher temperatures needed for brass and bronze.

Only in exceptional circumstances can electric furnaces compete with fuel-fired furnaces in melting brass, unless the alloy is one on which the use of an electric furnace can reduce the zinc loss, because of the high cost of electric power compared to fuel. Therefore it is evident that there is little hope of the electric furnace being able to compete with the oil-fired, iron-pot furnace in melting ingot aluminum, as ingot can be melted in pot furnaces with a loss of only one-third of 1 per cent, and there is no such margin for metal saving as in the case of brasses high in zinc.

When a large proportion of flux is to be melted, as in the fluxing method for aluminum chips, still more power will be required than in the melting of aluminum ingot. In the experimental furnace, and in most tilting electric furnaces, it is inconvenient or impossible to press the charge of chips and flux down into a compact mass, as is done in crucible melting. It is well known[b] that at temperatures below a red heat, a porous mass with inclosed air spaces is a good heat insulator and a poor heat conductor, so that notwithstanding the high heat conductivity of aluminum a mass of chips does not conduct heat very rapidly. Thus, if the layer of loose chips on the furnace hearth is thick, the top part of the layer, being nearest the arc, may be seriously overheated before the bottom part is hot enough to melt. From the salt fumes coming from the arc furnace when pouring, it appears probable that much salt is volatilized from the top of the layer, and that the chips on the top of the bath fail to coalesce and are so hot that they oxidize as soon as the furnace is opened to charge or to pour, and air enters. Hence much more flux is necessary than where the borings can be jammed down into a crucible, although in crucible melting with flux the constant stirring necessary in the puddling process is not required.

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[a] Richards, J. W., Electric power required to melt metals: Trans. Am. Brass Founders' Assn., vol. 4, 1910, p. 93.

[b] Ray, W. T., and Kreisinger, H., The flow of heat through furnace walls: Bull. 8, Bureau of Mines, 1911, 32 pp.

In an arc furnace the main source of heat is the arc itself, although some heat is of course reflected from the roof. As the arc is much hotter and much smaller than a flame, there is more danger of local overheating at the surface of the charge in an arc furnace than in a reverberatory furnace, where a sheet of flame covers the whole charge. An uncompacted mass of chips and flux, even at 900° C., is so viscous that rocking the arc furnace back and forth fails to stir the mass enough to mix it and prevent local overheating.

Hence, although there is a possibility that in a suitably lined electric furnace, where the lining is not attacked by the flux (linings of magnesite brick or mixtures of graphite and fire clay might perhaps serve) aluminum borings could be melted on a larger scale than in iron pots or crucibles, the reduction in labor cost would probably be more than offset by the higher cost for power as compared with the cost for fuel in ordinary types of furnaces.

EXPERIMENT WITH GRAPHITE CRUCIBLE LINING.

Another run was made to determine whether the indirect arc furnace would give satisfactory results with a better lining, as regards losses, without using so much flux as in run 94. It was thought that the lining that had crumbled off had acted like so much dirt in the chips and required excessive amounts of flux.

As neither magnesite brick nor graphite and fire clay mixture was at hand, a No. 60 crucible was used as a lining for the furnace. A hole for the passage of the electrode was cut in the bottom of the crucible, and a hole for the charging door and pouring spout was cut in the side. The crucible was placed horizontally inside the furnace and fire clay was packed between the crucible and the former furnace lining and the hearth at the mouth end of the crucible, the packing being completed with a mixture of ground crucibles and enough fire clay for a bond. This reduced the capacity of the furnace very greatly.

Run No. 96.—After the furnace had been preheated with a power consumption of 12 kilowatt-hours, about two-thirds of a charge of 8 pounds of chips mixed with 2.4 pounds of flux consisting of 85 parts NaCl and 15 parts CaF_2 was added. After 15 minutes, when 8 more kilowatt-hours had been used, the other third was charged, and after heating 5 minutes longer, 3 kilowatt-hours being used, the charge was at 975° C. The flux was only pasty and some thermit-like action was noted on top of the charge. Three and seventy-five hundredths pounds ingot and 1.25 pounds buttons were obtained, or a primary yield of 47 per cent, and a total yield of 62.5 per cent, on the basis of chips charged. As the crucible used as lining was not attacked and the result of this run was poor, the poor results of previous runs were evidently not mainly due to the crumbling of the lining.

Although run 94 indicated that with a sufficient amount of flux, electric melting in an indirect arc furnace might be possible, it seemed very unlikely that it would be commercially profitable and experiments with the indirect arc furnace were discontinued.

EXPERIMENTS WITH CRUCIBLE, RESISTOR FURNACE.

As lot D was from chips on which the commercial recovery was known to be 77 per cent on the weight of the chips, it was decided to make a few crucible melts to compare the experimental results with the commercial ones.

For these melts a No. 10 crucible was embedded in the granular graphite resistor of an electric resistor furnace, the crucible being taken out for pouring.

Run No. 97.—The crucible was first heated to dull redness, then small amounts of borings were added from time to time and puddled, care being taken not to let the temperature rise appreciably above the melting point. It took 10 minutes thus to work in 3 pounds of borings. Then the crucible was covered and heated 3 minutes more, when 2 per cent of fused $ZnCl_2$ was stirred in, the dross skimmed into water, and the metal poured. The dross showed a little thermit-like action. Two and twenth-five hundredths pounds ingot and 0.5 pound buttons was obtained, or a primary yield of 75 per cent and a total yield of 77 per cent, on the basis of the weight of the chips—that is, the same recovery was made as was found commercially.

Run No. 98.—Borax, which has been suggested as a flux for aluminum chips, not having been previously tried, 3 pounds of chips and 0.75 pound of pulverized crystalline borax, equivalent to about 0.4 pound borax glass, were mixed together and most of the mixture was charged into the cold crucible. The furnace was then heated and after 16 minutes the rest of the charge was added and the whole poked continually as it melted down, to approximate conditions in the puddling process. After 7 minutes more the charge was heated to more than 1,000° C. but the borax flux was only pasty, and mostly at the bottom of the crucible. The ingot recovery was 2.1 pounds and there was 0.20 pound of buttons, giving a primary yield of 70 per cent and a total yield of 76.5 per cent on the basis of chips charged. There was no thermit-like action.

Run No. 99.—As it seemed possible that the use of a larger amount of borax, with the addition of some fluorspar, might give a more fluid flux, the next run was made as follows: Three pounds of chips mixed with 1.2 pounds of pulverized crystalline borax and 0.1 pound fluorspar were charged into the hot crucible. In 16 minutes the temperature was over 1,000° C., but the aluminum was all on top and the flux all on the bottom. Only 1.3 pounds ingot, or 43 per cent primary recovery, was obtained. The mass of flux in the bottom of the crucible was full of exceedingly minute globules of metal, which were not recovered. Borax is evidently too heavy to serve as a good flux.

Run No. 100.—Five pounds of chips was mixed with 1.5 pounds of the flux, consisting of 85 parts NaCl and 15 parts CaF_2, and as much as possible of the mixture was packed into the cold crucible. After 20 minutes heating the rest was added, and after a total of 49 minutes the charge was at 950° C. The flux was quite fluid. Three and seventy-five one-hundredths pounds ingot and 0.10 pound of buttons were obtained, or a primary yield of 75 per cent and a total yield of 77 per cent on the basis of weight of chips taken.

Run No. 101.—Five pounds of chips was washed 10 minutes with 5 liters of 0.5 per cent NaOH solution, which removed much dirt, the NaOH was washed out, and the chips were stored under water 4 hours. They were then drained of water, mixed with 1.5 pounds of the flux, consisting of 85 parts NaCl and 15 parts CaF_2, and most of the damp mixture charged into the warm crucible. No oil smoked off, showing that it had been removed by the washing. After 15 minutes the rest of the charge was added, and after 15 minutes more the temperature was 975° C. The slag was fluid. Three and sixty-five one-hundredths pounds of ingot and 0.15 pound of buttons were obtained, or a primary yield of 73 per cent and a total yield of 76 per cent on the basis of weight of chips. There was a slight mechanical loss in washing and transferring the moist chips.

CONCLUSIONS FROM RESULTS OF TESTS.

The recoveries with the puddling method and the salt-fluorspar flux method, the latter on both dirty and washed chips, were practically identical and the same as the commercial recovery on these chips—that is, about 77 per cent on the weight of the chips and 87.5 per cent on the metallic content. As the use of salt-fluorspar flux had given better results on previous lots of chips in a gas furnace than the puddling method, it seems likely that the previous poorer results of the puddling method may have been largely due to the fact that the products of combustion in the gas furnace were continually sweeping over the metal as it was poked in. In run 97 there were no products of combustion, and the conditions were very like those in the iron-pot furnace.

It appears that, as puddling can not be done efficiently in a pit furnace, the use of salt-fluorspar flux is the most promising method of melting chips in pit furnaces. The temperatures required in the flux method are too high for the iron-pot furnace, and in that furnace the puddling method is preferable. In reverberatory melting the flux method is probably the better.

The choice of the three methods—puddling in the iron-pot furnace, use of the flux in pit or tilting crucible furnaces, or of the flux in a reverberatory furnace—for commercial refining can not be decided by laboratory tests. Costs of fuel, flux, crucibles or iron pots, furnace repair, and labor, the output in regard to floor space and furnace cost, the yield of ingot, the quality of ingot, the ease of recovering buttons from slag or dross, and other factors must all be taken into account in making a choice. It seems likely that for very clean chips the iron-pot puddling process would be more economical, whereas for very dirty chips the flux methods, whether in crucibles or in a reverberatory furnace, would apparently have a slight advantage.

QUALITY OF INGOT FROM ALUMINUM CHIPS.

Ingot made from dirty aluminum borings or chips is necessarily somewhat less pure and less desirable than virgin metal. One would hesitate to use all boring ingot in important castings. However, in small castings, such as are molded on the bench, if they do not have to stand a pressure test, 10 per cent of good boring ingot can usually be added to 90 per cent of new metal and back gates without increase in the proportion of defective castings and without detriment to the quality of the castings. In the case of large castings of complicated design, it is a moot question among aluminum founders whether or not the use of boring ingot increases the amount of castings defective through cracking in the mold. In some cases which have come to the attention of the writers, boring ingot has given very low "hot shortness" tests, which would indicate a liability toward cracking in the mold. On the other hand, some foundry men have claimed that certain boring ingot, used in quantities even up to 30 per cent of the weight of the charge, markedly decreases the loss from cracking, although other ingot of practically the same composition markedly increases it. The matter is a difficult one to settle, as so many factors, such as the relative hardness or softness of the cores, the ramming of the mold, the temper of the sand, the design of the casting, the gating, and the temperature at which the metal is poured, may all have as much or more effect on the tendency to crack as has the composition of the metal. In fact, the analysis of the metal gives no real criterion as to its tendency to crack.

However, it is true that some metal does crack worse than other metal of the same composition. Some unpublished results of work by H. Goldberg have indicated that metal melted under conditions where waste gases from a strongly oxidizing flame may reach it is more likely to crack than would the same metal if melted under reducing conditions.

Although boring ingot does not command the price of new metal, a boring ingot not too high in impurities finds a ready market at a price not much below that of new metal, and this is the best indication that there are plenty of castings in which boring ingot may be satisfactorily used.

On the other hand, there has been some boring ingot on the market, evidently made from a miscellaneous mixture of aluminum, babbitt, and other white metal and brass and bronze chips, which resembles aluminum only in its color, and which is of very little value to the founder. For example, two samples of metal offered for sale by one smelting company were analyzed by the foundry buying them, with the following results:

Composition of boring ingot made from various alloys.

		Sample A.	Sample B.
Copper	per cent..	17.4	15.3
Zinc	do....	8.4	6.7
Iron	do....	1.8	2.0
Silicon	do....	2.4	2.8
Manganese	do....	0.5	Trace.
Aluminum	do....	69.5	73.2

Samples of boring ingot from another company, sold as G. M. B. (good merchantable brand) metal, not as any standard alloy, analyzed by the buyer, gave results as follows:

Composition of G. M. B. metal.

Sample No.	Copper.	Zinc.	Iron.	Silicon.	Lead.	Tin.	Aluminum.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
C	6.4	25.0	1.0	0.3	1.9	1.3	64.1
D	6.8	6.0	0.7	0.2	1.3	0.6	84.4
E	7.5	5.6	0.8	0.4	2.0	1.0	82.7

Another sample (F) from this firm, sold as No. 12 boring ingot, analyzed by the buyer, contained 7.2 per cent copper, 3.9 per cent zinc, 1.5 per cent iron, 0.4 per cent silicon, no lead nor tin, and 87 per cent aluminum.

Samples of boring ingot from a third company (all sold on the seller's analysis), melted in crucibles in coke fires with NH_4Cl flux, had the following compositions:

Composition of boring ingot melted in crucible coke furnace with NH_4Cl flux.

Sample No.	Copper.	Zinc.	Iron.	Silicon.	Manganese.	Aluminum.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
G	4.2	2.5	9.3	0.6	1.2	82.2
H	3.9	12.1	3.0	0.5	0.15	80.35
I	7.3	4.3	1.5	0.4	....	86.5
J	7.7	0.3	2.8	1.9	....	87.3
K	8.8	0.75	2.0	1.0	....	87.45
L	9.4	1.0	1.3	0.3	....	88.0
M	7.7	0.3	2.8	1.9	....	87.3
N	9.7	0.7	1.1	1.0	....	87.5

Three samples taken from various parts of a quantity of boring ingot offered for sale by a fourth company, and analyzed by the buyer, had the following composition:

Composition of three samples of boring ingot.

Sample No.	Copper.	Zinc.	Iron.	Silicon.	Aluminum.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
O	7.9	2.0	0.6	0.4	89.1
P	7.8	0.4	.7	.4	90.7
Q	7.9	2.0	.7	.4	89.0

The results of buyers' analyses of various lots of No. 12 boring ingot sold by a firm using the iron pot, $ZnCl_2$, puddling process, were as follows:

Composition of No. 12 boring ingot.

Sample No.	Copper.	Zinc.	Iron.	Silicon.	Aluminum.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
R	7.2	1.5	1.3	0.2	89.8
S	7.5	1.0	1.3	.3	89.9
T	7.6	1.5	1.5	.2	89.2
U	6.6	1.5	1.3	.2	90.4

Still another refining plant, using a reverberatory furnace and dealing with a mixture of No. 12 chips and emery grindings, has sold No. 12 boring ingot of the following composition, according to the buyer's analysis:

Composition of ingot from No. 12 chips and emery grindings.

Sample No.	Copper.	Zinc.	Iron.	Silicon.	Aluminum.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
V	7.3	2.4	1.3	0.2	88.8
W	8.6	0.4	1.1	.2	89.7
X	8.8	0.8	1.4	.4	88.6
Y	9.6	0.9	1.0	.4	88.1
Z	9.9	1.0	1.1	.3	87.7

The No. 12 alloy, made up from new metals, to contain 8 per cent copper, or purchased in market ingot made up from new metals, will analyze about 7.6 to 8.4 per cent copper, no zinc, 0.5 to 0.7 per cent iron, 0.25 to 0.4 per cent silicon, 91.65 to 90.5 per cent aluminum.

When melting in iron pots, about 0.2 per cent of iron is taken up from pots, ladles, and skimmers and about 0.05 per cent silicon from sand adhering to gates, making the average composition of the No. 12 castings sold 7.6 to 8.4 per cent copper, no zinc, 0.7 to 0.9 per cent iron, 0.30 to 0.45 per cent silicon, and 91.4 to 90.25 per cent aluminum. This alloy comprises 90 to 95 per cent of all cast aluminum made in the United States.

EFFECT OF IMPURITIES.

The purity, or impurity, of boring ingot depends (1) on the purity of the castings from which the borings are made, (2) on the freedom of the borings from white metal, babbitt, or brass, (3) on their freedom from iron and on the efficiency of the magnetic separation, (4) on the presence or absence of dirt from which metals may be reduced, and (5) on the nature of the melting process and the temperature attained.

White metal, babbitt, brass, or bronze borings, if present, can not be efficiently separated from the aluminum borings and go into the melt. Copper, zinc, lead, tin, and antimony may come from these sources.

COPPER.

Of these metals copper is present not only in the No 12 alloy, but also in nearly all of the zinc-containing alloys, and its only bad effect would be to raise the copper content too high.

ZINC.

Zinc in No. 12 castings is frowned on by many purchasers, even if present only to the extent of a trace to less than one-half of 1 per cent, in some cases because it is known that zinc can not occur in No. 12 metal made wholly from virgin metal, but more often because a belief that the alloys of aluminum high in zinc give castings which may break in use under repeated shock is extended to the No. 12 alloy with but traces of zinc. This idea is prevalent in some sections of the country, whereas in other sections the zinc-containing alloys are much used. Alloys, such as the market "No. 31," containing about 3 per cent copper, 15 per cent zinc, 0.5 per cent manganese, 81.5 per cent aluminum, or a similar one containing 2 per cent copper, 19 per cent zinc, and 79 per cent aluminum, are stronger under tension or compression per unit weight than the No. 12 alloy, and, at normal prices for zinc, are cheaper per unit of weight or of volume. It does not appear to be satisfactorily proved that perfectly sound castings of these alloys are more likely to fail under repeated stress than the No. 12 alloy, as an alloy containing 19 per cent zinc has been satisfactorily used in one high-grade American automobile, and zinc-aluminum alloys, somewhat lower in zinc content, are used in European automobile plant practice almost to the exclusion of the No. 12 alloy, whereas the reverse is the case in the United States. It is claimed that because of the rougher roads in America the stresses a motor-car crank case, for example, has to endure are worse than in Europe, and that the zinc-containing aluminum alloys would not stand up as well as the No. 12 alloy under the more severe conditions in America. It is probable that the relative life of the crank cases and other castings on the American and European motor cars and trucks in the European war will furnish some definite proof on this point.

It is true that it is more difficult to make complicated castings of zinc-containing alloys than of No. 12 alloy without having trouble from cracking in the mold, and that if castings with tiny cracks in them that have been undetected through lax inspection are put in service, failures are likely to result. But it is quite possible to make distinctly complicated castings of the zinc-containing alloys without defects if proper foundry practice is used.

Rosenhain and Archbutt [a] have made endurance tests of various aluminum alloys and do not find that the zinc-containing alloys are liable to fail under repeated shock. They indeed advocate the use of an alloy consisting of 20 per cent zinc and 80 per cent aluminum, or one containing 3 per cent copper, 25 per cent zinc, and 72 per cent aluminum, rather than those lower in zinc content, on account of the greater strength per unit weight.

A mixture of aluminum borings from various sources in the United States will consist mostly of No. 12 alloy, but may also contain some borings from alloys containing 10 to 30 per cent zinc, consequently the ingot from such mixed borings may contain zinc in varying amounts, as is shown by the analyses previously given.

As zinc volatilizes quite readily from aluminum alloys at the temperatures necessary in melting chips, some of the zinc content of alloys high in zinc will be lost in melting. A few hundredths of a per cent of zinc in No. 12 alloy might come from the taking up of zinc from $ZnCl_2$ used as a flux, whereas a few tenths of a per cent or more of zinc indicates that some scrap, or else No. 12 ingot made in part from scrap, has been used. There is no satisfactory evidence to show that a zinc content up to 0.5 per cent at least in No. 12 alloy is any detriment to the strength or endurance of the castings. As the presence of zinc indicates the presence of scrap and presumably of boring ingot which may, under conditions not clearly understood, give rise to the cracking of castings in the mold or to other foundry defects, some progressive founders are using virgin pure aluminum and copper and preparing their own No. 12 alloy instead of purchasing the prepared alloy, and are using analyzed boring ingot in varying proportions, or leaving it out altogether, according to the liability of the individual casting that is to be poured from the metal to the troubles which boring ingot sometimes causes. With such rational utilization of boring ingot it finds a legitimate use in the foundry.

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[a] Rosenhain, W., and Archbutt, S. L., Alloys of aluminum and zinc: Engineering, vol. 93, 1912, p. 578.

LEAD.

Lead is not a desirable constituent in aluminum alloys, as it does not alloy with aluminum, but is merely inclosed in small globules. The lead, on account of its high specific gravity, will tend to collect at the bottom of the casting, and any appreciable amount tends to weaken the alloy. Such boring ingot as that shown by analyses C, D, and E is of poor quality on account of the lead content.

TIN.

Tin is to be classed as a less harmful impurity. It tends to soften the alloy without noticeably affecting the strength. A small producer of high-grade automobiles has used an alloy containing 5 per cent copper and 3 per cent tin without any apparent disadvantage save a slightly lower strength. Such an alloy made from new metals has no obvious advantage to counterbalance the high cost of the tin, but a few tenths of a per cent of tin is not to be feared in boring ingot, save that, as it usually comes from admixed solder or from bronze chips, it may be accompanied by lead.

ANTIMONY.

The effect of antimony on aluminum alloys is not well known. Its occurrence in boring ingot would indicate admixture of babbitt with the chips.

MANGANESE.

Manganese in appreciable proportion would indicate that the ingot was made from material containing more or less hard sheet clippings. In small amount, with a high zinc content, for example, in analysis H, it would indicate that the mixture melted contained some No. 31 alloy chips.

In percentages up to say 0.2 per cent, manganese is not a detriment to No. 12 alloy, and some founders think that 0.1 or 0.2 per cent is distinctly desirable, although manganese is seldom intentionally added to No. 12 alloy, its source being some form of scrap from hard-sheet aluminum.

IRON.

Iron is the main impurity in boring ingot made from No. 12 chips and comes from the admixture in the machine shop of iron or steel chips with the aluminum chips. Commercial magnetic separation of iron from such chips may leave, especially in the case of very oily chips, 0.5 to 1.0 per cent of free iron in the mass. All of this free iron goes into solution when the mass is melted. Save for the free iron, no increase in iron content would be shown in melting chips in crucibles and not over 0.25 per cent increase in melting in iron pots by the puddling process.

INTERRELATED EFFECTS OF IRON, COPPER, AND ZINC.

As high as 2 per cent of iron in the No. 12 alloy would probably give too brittle an alloy, but a content of between 1 and 1.5 per cent does not appear to have a notably detrimental effect. If the iron content rises above 1 per cent, the copper content should be correspondingly reduced below 8 per cent in order to prevent brittleness, as iron acts much like copper in hardening and strengthening aluminum.

Cast No. 12 alloy, unannealed, consists of a needle-like $CuAl_2$–Al eutectic embedded in the groundmass of solid solution of copper in aluminum. Iron in aluminum causes a similar needle-like structure in a groundmass of solid solution, and as the properties of the No. 12 alloy depend largely on the proportions of needles and groundmass too high an iron content will give too much of the needle-like material. If zinc be added to the No. 12 alloy, more of the needle-like eutectic is thrown out than without it, so that in the commercially useful ternary alloys of copper, aluminum, and zinc the copper is used in smaller amount as the zinc is used in larger amount. For example, an alloy containing 7.5 per cent copper and 1 per cent zinc has about the same tensile strength and elongation as one containing 8 per cent copper. With 20 per cent zinc not over 4 per cent copper and with 30 per cent zinc not over 2 per cent copper should be used, else too brittle an alloy will result.

By proper reduction in the copper content the effect of high iron content and of a little zinc, introduced by the use of some boring ingot, can largely be neutralized, and the resulting alloy will still have very closely the properties of No. 12 alloy as regards foundry behavior, shrinkage, and tensile strength and elongation.

SILICON.

Silicon is generally considered to be an undesirable impurity and, especially if in the form of graphitic silicon, to be conducive to brittleness. It is always present in small amount in commercial aluminum, owing to the difficulty of completely freeing from silica the bauxite used in its manufacture.

In remelting gates to which molding sand adheres, or in melting chips that contain siliceous dirt some silica may be reduced to silicon which alloys with the aluminum and an equivalent amount of aluminum is oxidized and lost.

This reduction of silica by aluminum is not marked below 800° C. and even when melting in fire clay graphite crucibles only a very small content of silicon is taken up if the temperature is kept low. As the temperature increases reduction takes place more readily, so that at the temperatures necessary in melting chips some slight increase in silicon content is to be expected if they contain siliceous dirt. The use of a flux that will dissolve or soak up the dirt and so take it out of contact with the metal might be expected to decrease the amount of silicon taken up at any given temperature.

The results of analyses of commercial lots of boring ingot as shown on pages 73 and 74 give no information as to the relative purity of ingot made from the same lot of chips by different methods of melting, as any such differences are doubtless overbalanced by variations in the purity of the alloys from which the chips were taken and by variations in the content of free iron and dirt.

COMPOSITION OF INGOT FROM ELECTRIC FURNACE.

Analyses were made by the writers of the ingot obtained in the 15-pound melts of chips from lot B in the electric crucible furnace, in run 69 when a yield of 58 per cent on the original weight of the borings was obtained with sal ammoniac flux, in run 70 when a yield of 71.5 per cent on the original weight was obtained with NaCl–CaF$_2$ flux, and in run 83 when a yield of 89 per cent was obtained with the NaCl–CaF$_2$ flux on the fraction over 14-mesh in size. The results of the analyses were as follows:

Composition of ingot from melts in electric furnace.

Run No.	Copper.	Iron.	Silicon.	Manganese.	Zinc.	Aluminum (by difference).
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
69	7.40	1.87	0.34	Trace.	None.	90.39
70	7.39	1.92	.36	Trace.	None.	90.33
83	7.42	1.25	.34	Trace.	None.	90.99

The only appreciable difference is in the lower iron content of the fraction over 14-mesh, which indicates that most of the free iron left in this sample after commercial magnetic separation was under 14-mesh, an assumption which is corroborated by visual examination. Notwithstanding the difference of 13.5 per cent in the yields of runs 69 and 70, the composition of the ingot obtained is identical. As the run with the salt and fluorspar flux shows no appreciable increase in silicon content although the borings contained over 12 per cent of siliceous dirt, and as, unless the borings are melted in an iron pot, the iron content can not be greater than the original content in the alloy from which the chips were made plus the free iron not removed by the magnetic separator, the criticism that the high temperature needed to make this flux fluid results in ingot higher in impurities than by other methods of melting does not seem well founded.

NECESSITY FOR CARE AND CLEANLINESS IN COLLECTING CHIPS.

The high loss in melting chips caused by difficult coalescence and subsequent oxidation of globules entangled in the dross, and the poor quality of boring ingot are due primarily to lack of care and cleanliness in the collection of the chips in the machine shop. If the aluminum chips are kept free from brass, bronze, solder, white-metal, or iron chips, and also free from dust and floor sweepings, the only foreign material in them will be the cutting oil or cutting lubricant. By promptly centrifuging the chips these lubricants can be removed and recovered, and the chips left in a better condition for melting. If chips are thus collected and centrifuged and then stored in covered receptacles so that dust can not settle on them, clean chips will result.

The value of clean chips is so much greater than that of dirty chips, mixed with iron chips, that the slight increase in cost of careful collection over that of collecting by slipshod methods would be many times repaid and on the large amount of chips produced by even a comparatively small manufacturer of motor cars the saving would be well worth while.

As at least two methods of melting chips now in use by refiners, the puddling process and the $NaCl-CaF_2$ process, will give a recovery of 90 per cent or higher on absolutely clean chips and produce boring ingot of good quality, the responsibility for the waste of aluminum in melting chips, which is large in the aggregate, rests at the door of the machine shop that permits iron and dirt to get into the chips, rather than at the door of the foundry or refining plant that attempts to turn the chips into usable ingot.

Foundry refuse, such as buttons recovered from skimmings can not, in the nature of the material, be collected free from foreign matter. Hence such material, as well as carelessly collected chips, offers difficulties in melting owing to reluctance of the globules formed in melting to coalesce.

PROBABLE RELATIVE USEFULNESS OF DIFFERENT MELTING METHODS.

Of the methods suggested for melting aluminum chips, some are of doubtful practicability. Feeding the chips into the molten electrolytic bath used for producing pure aluminum is plainly useless in the case of dirty chips, on account of fouling the rather expensive electrolyte. The method is not well understood by founders and refiners, as the details of the electrolytic process are kept secret by the producers of aluminum, so that even on clean chips the method would be of use only to those producers.

Briquetting involves the use of expensive machinery, and although the use of briquets might slightly decrease the melting loss, would probably materially decrease the melting time and fuel cost, and would certainly increase the ease of handling, yet it is likely that a briquetting machine of the type necessary to produce solid briquets would have to be kept in constant use in order that the interest on the first cost should not eat up the savings.

Hence briquetting would only be practiced by large refiners who have large amounts of aluminum chips to handle or have other uses for such apparatus. Even at such plants the question whether briquetting would pay is not yet fully answerable, although the process deserves serious consideration.

The use of electric furnaces, vacuum furnaces, retorting furnaces, or other devices which merely prevent the access of air without providing for the promotion of coalescence by stirring seems to offer little promise.

The method so often advocated of pushing the borings beneath a heel of molten metal seems to owe any usefulness it may have to the pushing and not to the heel. It is an approach to the puddling process, which seems to be the best modification of the scheme.

In the iron-pot puddling process, which is evidently of much value on clean borings, the vital points are to keep the temperature very low, practically at the melting point, to prevent oxidation, and constantly stir the pasty mass to promote coalescence. The process requires constant hand labor, does not seem as useful on very dirty borings, and could hardly be applied to large units.

Possibly some mechanically stirred furnace, or one in which pressure was constantly applied, analogous to the Côte and Pierron suggestion or to the Montefiore method for melting blue powder, could be devised that would have the advantages of the puddling process without the high labor cost, but no such apparatus had yet been put forth, and the making of one that would have a low upkeep cost presents serious difficulties.

The use of volatile fluxes such as sal ammoniac and zinc chloride is of advantage at the end of the puddling process, but melting with these fluxes without constant stirring gives very low recoveries.

The use of fluxes that form molten covers seems to be of real advantage only when rather large quantities of the flux are used, and the promotion of coalescence by the flux soaking up dirt and oxide seems to be of more importance than the prevention of oxidation by exclusion of air. Although fluxes melting at or below the melting point of aluminum are theoretically preferable to those of higher melting point, it is difficult to find such low-melting fluxes chemically inert to aluminum without using mixtures containing materials such as lithium chloride or potassium chloride, the first of which is very costly and the second relatively so, or hygroscopic materials such as calcium or magnesium chlorides. Common salt, the cheapest flux of the molten-cover type, seemingly gives the best results, when the cost and convenience of the flux is considered. Still better are fluxes that have a real solvent action on aluminum oxide and on dirt in general, such as the mixtures of alkali chlorides and fluorides. With such fluxes better absolute recoveries could probably be obtained by using very low melting mixtures, but as large amounts of these fluxes are necessary their cost is prohibitive, and the cheap flux consisting of 85 parts common salt and 15 parts fluorspar, used in large amounts, 20 to 30 per cent of the weight of the chips, mixed with the chips before charging, and the whole heated in crucibles till the flux is fairly fluid, seems the most useful. On clean borings it would probably not give higher results than the iron-pot puddling process, but it may be preferable even then as constant stirring is not required, and thus the labor cost would be less, and because it might make large-scale reverberatory melting feasible. The experiments on reverberatory melting indicated this possibility but were on too small a scale to be conclusive. If chips must be melted in pit fires, the flux method is the most suitable; if iron-pot furnaces are to be used, the puddling process is the better.

There is also a possibility that dirty borings washed with dilute caustic soda, subjected to wet concentration, as on Wilfley tables, and then dried by centrifuging, would yield a clean product on which the melting loss would be low. Lack of suitable apparatus made the actual experiments inconclusive, but the scheme seems worthy of a trial on a commercial scale.

However, if the machine shops producing aluminum chips would keep the borings clean and free from contamination, it would not be necessary to go to the expense of removing foreign matter.

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Bulletin 109

DEPARTMENT OF THE INTERIOR
FRANKLIN K. LANE, SECRETARY
BUREAU OF MINES
VAN. H. MANNING, DIRECTOR

OPERATING DETAILS OF GAS PRODUCERS

BY

R. H. FERNALD

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OPERATING DETAILS OF PRODUCER-GAS INSTALLATIONS.

By R. H. Fernald.

INTRODUCTION.

In 1900, as far as available records show, there were only two producer-gas power installations in the United States.

In June, 1915, the number probably exceeded 1,000. Of this number, some 84.5 per cent comprised small plants using anthracite, and only about 15 per cent of the total number utilized bituminous coal and lignite. In addition to these, two plants used wood, and three or four used oil.

In the main, however, the larger sized units are operating on bituminous coal and lignite.

During the early years of these plants little success was anticipated from the use of bituminous coals and lignites, but in 1904 and 1905 it was fully demonstrated at the Government testing station at St. Louis that these fuels could be utilized to excellent advantage. However, although many producer-gas plants in daily operation are using bituminous coal and lignite, many power-plant men are still skeptical regarding the successful commercial use of these fuels.

PRESENT STATUS OF PRODUCER-GAS PLANTS.

In this connection the author can offer no clearer idea of the present status of the producer-gas power plant than is presented in his letter of March 4, 1915, to the prime movers committee of the National Electric Light Association in response to general queries from the committee. The text of the letter follows:

In response to your request for a brief summary of the producer-gas situation, I take pleasure in submitting herewith a brief outline of some of the more important phases of this development and in answering the queries that you have put to me.

1. *General classification of gas producers.*—Producers for power purposes can be broadly classified as (*a*) up-draft, (*b*) down-draft, (*c*) double-zone.

In the up-draft plant the pressure in the gas generator may be greater or less than that of the atmosphere. If it is greater than that of the atmosphere, the plant is known as an up-draft pressure plant. If it is less than that of the atmosphere, the plant is commonly called an up-draft suction plant, although this reduction of pressure in the gas generator of the larger plants is now usually produced by means of an exhauster instead of by the suction stroke of the engine.

2. *Producers for anthracite coal.*—Little difficulty has been experienced in handling good grades of anthracite coal in gas producers. Occasionally some trouble is experienced owing to the character of the ash or to a low ash-fusing temperature. In the main, however, this fuel has been found satisfactory. For most sections of the country the price of anthracite is relatively too high to warrant its use in plants of large capacity. It is, therefore, largely utilized in plants not exceeding 500 horsepower. As far as I know little has been done in this country with gas producers for the utilization of anthracite screenings or material from the culm pile.

Anthracite coal may be utilized to good advantage in plants of either the up-draft or the down-draft type. Inasmuch as it is comparatively free from tar, anthracite is commonly used in the up-draft producer of either the suction or the pressure type. A single installation of 4,000 horsepower of down-draft producers is using anthracite at $11.30 a ton in preference to bituminous coal for which the plant was designed. Although the company owns bituminous mines, it places a value of $8 a ton on its books for the bituminous coal. On this basis of $8 a ton for the bituminous coal and $11.30 a ton for the anthracite, a year's operation shows financially in favor of the anthracite. Outside of two or three installations, the individual anthracite plants of this country do not exceed a few hundred horsepower.

3. *Bituminous coal.*—Satisfactory gas producers have been designed for the use of both bituminous coals and lignites of good quality. There is comparatively little difficulty in handling on a commercial scale such plants, provided the fuel is low in ash, has a fairly high ash-fusing temperature, and does not give serious trouble from caking and clinkering. Unfortunately these restrictions are too exacting to fit our common practice in the United States with low-priced fuel. The European situation, where they are able to specify rather definitely the characteristics of the coal, is very different.

My answer, therefore, to your query as to whether producers have been successfully designed for the use of bituminous coals and lignites is "yes" for bituminous coals and lignites of high grades.

I do not say "no" for other grades of bituminous coals and lignites, but I realize that low-grade fuel, high in ash and prone to clinker troubles, is not regarded in the majority of cases as worth the time and effort required. Bituminous coals and lignites of good grade may be successfully used in the up-draft producer if adequate equipment is installed for scrubbing the gas and removing the tar, in the down-draft producer of the continuous type, and in the double-zone producer.

The largest single generator in the United States with which I am familiar is one of 250 square feet of fuel-bed area, burning between 3,000 to 4,000[a] pounds of Illinois bituminous coal per hour. I see no reason why single-shell producers of this type should not be built four times this capacity.

4. My estimate of the horsepower capacity of gas producers installed in the United States for power purposes is as follows:

For anthracite coal:	
Plants of more than 500 horsepower rating, horsepower	40,000
Plants of less than 500 horsepower rating, horsepower	95,000
For bituminous coal, horsepower	130,000
For lignite, horsepower	15,000

My estimate of the annual fuel consumption of these plants is, roughly:

Anthracite coal, short tons	240,000
Bituminous coal, short tons	400,000
Lignite coal, short tons	60,000

---

[a] These figures were reported at the time of making the installations. (The latest reports (June, 1915) give the fuel-bed area as 210 square feet and the average fuel consumption as 2,750 pounds per hour.

5. In response to your query regarding the slow progress in the development of the bituminous-coal producer, I name the following as among the reasons that immediately suggest themselves:

(*a*) The low price of our fuels. The time has seemingly not yet arrived when there is sufficient demand for reduced operating costs to warrant the investment necessary for the production of producer-gas units of sufficient size to seriously compete with the efficient steam-turbine units of the present day.

(*b*) As a corollary to *a*, the cost of the labor involved and the reduced capacity limit producers to the utilization of fuels of good grade. As soon as the cost of fuel is sufficiently high to warrant the use of high ash, low-grade material the design of a producer for this purpose will be speedily forthcoming.

(*c*) Additional difficulties are satisfactory gas cleaning, tar removal or recovery, elimination of sulphur, and the prevention of serious clinkering. All of these difficulties can, I believe, be obviated.

(*d*) The fact that, up to the present time, no gas engine has been developed of sufficient size to enter into serious competition with the recent steam-turbine units of 30,000 kilowatt and 35,000 kilowatt capacity.

PRODUCER-GAS PLANTS IN EUROPE.

In considering the many phases of this problem you will, I believe, be interested to know the trend of European producer-gas practice to-day. I therefore append a few notes gleaned from my inspection last summer in the interest of the United States Bureau of Mines.

We find in Europe a demand for a gas producer to handle all grades of fuel, especially those grades usually sent to the dump. This demand has brought to the European market the revolving, eccentric-grate producer. Among the most important advantages claimed for these producers is automatic ash removal. Dependent on this primary advantage rest the following claims for the revolving grate:

1. Low labor cost for handling ashes.
2. More uniform and more complete combustion.
3. Operation for months without interruption.
4. Ability to handle much more fuel per square foot of fuel-bed area.
5. Less space per 1,000 cubic feet of gas produced or per horsepower of plant.
6. Freedom from dust and the usual excessive heat and dirt during removal of ashes.
7. Production of a gas of nearly uniform quality.
8. Reduction in the cost of upkeep.

If in addition to rotating the grate the grate be placed slightly off center, a feature is introduced that is probably of far greater value in handling high-ash, clinkering fuels than the mere rotation of the grate.

Experience with European fuels has shown that even with the eccentric revolving grate and the usual producer shell construction clinker troubles are not entirely eliminated when poor-grade fuel with low ash-fusing temperature is used. A further important feature—probably the most important single item—for overcoming clinkering and the tendency of the ash to fuse with the producer lining is the water jacketing of the part of the producer shell surrounding the hot zone.

These revolving grate producers are reported to gasify two to three times as much fuel per square foot of fuel-bed area per hour as can be gasified in corresponding updraft producers with fixed grates.

Claims of very low percentages of carbon in the ash are also made for this type of producer, the reported record for one installation being 5 per cent carbon, or 0.47 per cent of the fuel gasified.

The claims advanced regarding the steam requirements for clinkering coals used in producers with water jackets around the hot zone are to the effect that not over one-quarter as much steam is required as in the jacketless type with fixed grate. The figures given for comparison are 1 pound of steam per pound of fuel for the fixed-grate jacketless producer and 0.29 pound for the revolving eccentric-grate producer. Results with United States coals in fixed-grate jacketless producers indicate that 1 pound of steam per pound of coal is rather high for plants of good size. Seven-tenths of a pound is nearer the figure, although there are undoubtedly many plants, indifferently operated, that are not below the 1-pound rate.

Representatives of companies handling eccentric revolving-grate producers say that they handle coal containing 45 to 55 per cent ash with perfect ease and are satisfied that these producers can meet the conditions required for American high-ash caking coals.

In Europe we also find a great deal of attention given to the by-product gas plant. These plants are not of a few horsepower capacity, but the installations range from 5,000 to 30,000 horsepower. One company alone reports the installation of by-product recovery producer-gas plants using a total of 3,000 tons of fuel per day and aggregating approximately 300,000 horsepower. The capacity and purpose of a few of the larger installations are as follows:

Capacity and purpose of a few of the larger European producer-gas plants.

Installation No.	Fuel capacity per day.	Purpose of plant.	Installation No.	Fuel capacity per day.	Purpose of plant.
	Tons.			*Tons.*	
1	320	Recovery of by-products from waste fuels. Gas used for firing boilers and for power.	5	135	Power, forge, and plate furnaces, fire-clay kilns, etc.
			6	125	Power and firing caustic pots.
2	270	Central distributing station.	7	120	Evaporating brine
3	250	Power and chemical purposes, calcining ore, etc.	8	120	Power and chemical furnaces.
			9	100	Firing chrome furnaces.
4	150	Special plant for the recovery of by-products. Gas used for firing colliery boilers.	10	100	Chemical furnaces.

The majority of these plants are used for power development and gas heating; the recovery of by-products, such as sulphate of ammonia, tar, etc., are secondary factors in the operation of the plant. On the other hand, there are several installations in which power is the secondary factor, the plant being run primarily for the valuable by-product, sulphate of ammonia, which brings a commercial return of $50 to $60 a ton.

A few plants are operated for the by-products alone. In certain districts in which the manufacturing and industrial interests do not offer a market for the gas the so-called "by-products" become the main products and the true by-product, producer gas, is thrown away. This condition of affairs is peculiarly true in regions in which the fuel runs high in nitrogen. It is reported that an extensive plant of this character is soon to be erected in Africa.

Peat seems to be peculiarly adapted to the requirements for the production of sulphate of ammonia, and several commercial by-product plants using this fuel are now in operation in Europe. Among these are two plants in Italy using, respectively, 140 and 90 tons of peat per day.

One of the most interesting plants visited last summer was a by-product coke plant, in which the coke-oven gas was the main product and the coke the by-product, combined with a by-product producer-gas plant. The coke-oven gas was turned into the city mains for general use. In order to obtain all this gas for sale to the city, a distinct central producer-gas plant was installed, as the poorer grade gas, which was not available for general city use, was entirely satisfactory for heating the coke ovens. The originator of this combined method says: "The great advantage of this system is the fact that coke breeze and low-grade fuel generally can be used in the producers without lowering the efficiency of the plant." The producer plant consists of five units, each of 20 tons capacity a day. The coke from the ovens, which is regarded as a by-product, finds a ready market for blast-furnace work, and it is estimated that the by-products from the producer plant, sulphate of ammonia and pitch, practically pay the cost of operation of the producer-gas installation. The coal used in the producers is double-screened nut and contains no dust. It is a fine-looking coal and is reported to be high in oils.

The several companies manufacturing by-product gas plants believe that there is a large field for such plants in the United States if the fuels used be carefully selected. They regard our caking coals as bad for this type of plant.

SLAGGING GAS PRODUCER.

The blast-furnace type or slagging gas producer has appeared at regular intervals in Europe and investigations along this line have also been conducted in this country both by commercial interests and the United States Bureau of Mines. In spite of the many claims that no such producer can operate with any commercial success, we find at a colliery in Deutsch-Luxemburg a wonderfully interesting installation consisting of four slagging producers. These producers are approximately 10 feet in internal diameter and gasify 60 tons of fuel each per 24-hour day. The first of these producers was installed about two years ago and required much study and many changes. At the present time [March 4, 1915] these producers are working on 10 per cent ash fuel, but the company anticipates utilizing material containing 20 to 30 per cent ash. Several different companies are reported to be using these slagging producers, and the fact that this type of producer has a real commercial position is shown by the catalogue of one of the large producer manufacturers of Germany, which, among several other classifications, lists its producers as—

(1) Producers with rotating grates.
(2) Slagging producers.
(3) Flat-grate producers.
(4) Step-grate producers.

Considerable interest is also manifested abroad in the use of wood refuse and similar material in gas producers. At the present time, however, these plants are of small size.

The interest of engineers in Europe is also more or less centered on the possibilities of powdered fuel and questions relating to low-temperature distillation. The principal purpose of this latter problem is to procure a clean smokeless fuel for the unlimited number of fireplaces and at the same time to recover from the original fuel large percentages of oils, motor spirits, and ammonia.

In conclusion, I believe that the time is not distant when the price of our coals must necessarily materially increase. When this time does come we will look much more seriously on the important problems connected with our fuel conservation. We will find it imperative to use high-ash low-grade fuels which, in many cases, will have a low ash fusing temperature. Transportation of this high ash material will be out of the question and its conversion into gas at the mines will result. Even though large gas engines are not forthcoming, the economic use of grades of material that can not be utilized directly under steam boilers will warrant large installations for the production of producer gas and its use for steam generation for large turbo-generator units with long-distance electric transmission.

PRODUCER-GAS PLANT COMPARED WITH STEAM TURBINE.

The steam turbine naturally lent itself to central-station service. It was a unit easily understood by steam-engine operators in so far as practical operation is concerned; it could be readily erected without radical changes in the boiler-room equipment; and it rapidly met the demand for large central-station units.

The producer-gas plant was an untried factor; it met with strong opposition from the older steam-plant operators, who saw possibilities of being forced out of their positions; its installation meant a complete renovation of the entire plant, with the replacing of the steam boiler by the producer unit. Large units of this type did not materialize, with the natural result that even to-day the producer-gas power plant is not the large central-station unit, although it occupies a strong position among the isolated plants and the small central stations.

Methods of operation vary greatly with local conditions and especially with the personal inclination or opinion of the plant superintendent. This variation is, perhaps, perfectly natural, owing to the newness of producer-gas power, the absence of experienced producer-gas engineers, the absence of cooperation among the operators of these independent isolated plants, and the absence of well-defined commercial investigations.

With this lack of a common interest on the part of the owners or operators of these plants—an interest that has of late tended to place central-station operation on somewhat uniform basis—no standards for comparison of plant operations have appeared and little has been known by the superintendent of one plant regarding the relative efficiency of operation of his own installation and that of another plant of similar type operating under similar conditions.

Several unsuccessful attempts have been made by various organizations to collect data relating to these important matters, but unfortunately little or no information could be had.

Appreciating the value of such data in connection with the many problems of fuel conservation and its investigations into the use of producer-gas power, the Bureau of Mines decided (1) that an effort should be made to procure operating details from a small number of representative producer-gas plants using scrubbed gas either for power or heating purposes; (2) that primarily data from plants using bituminous coals and lignites should be sought, but that data from a few of the larger anthracite plants and the oil and wood plants should be included.

With these points in mind, data sheets were sent to the owners of a limited number of plants, and for the most part the response has been highly gratifying. Returns of a positive character were received from 39 installations.

Although, as might be expected, a great deal of indefiniteness exists regarding many of the operating details, and few, if any, cost data are available, it is believed that in the following pages sufficient detailed information is presented to provoke wholesome discussion on the part of those interested in this form of power; to stimulate responses to future requests from the Bureau of Mines, and to serve as a basis for comparison by the different operators of similar plants.

CHARACTER OF FUEL USED IN GAS PRODUCERS.

Owing to the agitation of the Bureau of Mines for several years past concerning the use of high-ash and low-grade fuels, it was hoped that several producer-gas installations would report the use of grades of fuel hitherto regarded as of little commercial value. However, an inspection of several plants in 1913 showed rather conclusively that on the whole the grade of fuel used in producer-gas installations has been steadily rising, and, in the main, the fuel used to-day is decidedly superior to that used six or eight years ago.

Although from the standpoint of the engineers of the Bureau of Mines this condition is to be regretted, it is perhaps perfectly natural, as the tendency of the operators of such plants is to reduce manual labor to a minimum. A feeling of assurance by the management of guaranteed reliability of service is also commercially important in determining the quality of fuel to be used. It is not strange, then, that the highest grade fuel obtainable at a reasonable price is, in nearly every instance, the one adopted.

A rather poor grade of bituminous coal was originally used in a plant of considerable size, but owing to the amount of labor required in operating, the purchasing agent was finally persuaded by the plant superintendent to substitute a high-grade coal at a much higher price per ton.

Such excellent results were at once obtained and the labor cost was so materially reduced that the purchasing agent, who had formerly believed in purchasing the cheapest coal to be had, immediately substituted the high-grade coal throughout the boiler plant as well as the producer plant. The superintendent felt that the greatest service the producer plant had rendered was in improving the grade of fuel used in the steam plant. He stated that he realized that his company was not working toward fuel conservation, but he felt that reliability of service was the uppermost requirement in his plant. He said that he would be glad to use a lower grade fuel at a less cost per ton if he could be shown plants with similar exacting commercial requirements successfully operating on such fuel.

Such comments are, for the most part, typical of the present situation. It is, however, gratifying to note here and there exceptions to this tendency. One notable example may be seen in Texas in a plant that uses lignite screenings as the regular fuel for the gas producers. The manager finds these screenings, costing much less than run-of-mine lignite, an excellent producer fuel. He has given a standing order for this material, but has considerable difficulty in procuring enough of it.

As far as is reported, no other operator in that vicinity feels any assurance of success with such screenings as they are generally regarded as inferior fuel.

The high character of the fuels generally used for producer-gas installations is made apparent by the data in the table following.

Grade of fuel used in different types of gas producers.

PRODUCERS USING BITUMINOUS COAL.

Plant No.	Ash.	Sulphur.	Heating value per pound as fired.
	Per cent.	*Per cent.*	*B. t. u.*
24	3.7	0.5	11,540
8	6.9	1.6	12,270
12	7.4	.5	12,296
17	10.1	3.4	12,522
23	6.7	2.2	13,741
28			13,750
37			14,000
33	7.0	1.5	14,370
39	5.0	.9	14,500
27	7.9	1.2	14,535
22	4.8	.5	14,769
3	7.3	.8	14,800
35	5.5	1.0	14,834
25	7.0	2.0	
20	4.7	1.0	

PRODUCERS USING LIGNITE.

11	7.9	1.0	5,560
4	6.9	.6	7,440
11	8.2	.4	7,070
31	6.0		11,400

PRODUCER USING WOOD.

26			5,400

Most lignite contains 30 to 40 per cent water, but, as indicated above, it does not necessarily run high in ash. As regards true fuel conservation, there is seemingly little being done in the United States by the operators of producer-gas plants.

TESTS OF LOW-GRADE FUEL.

It is, perhaps, important again to emphasize the possibilities from high-ash coals. Although it is fully appreciated that commercial conditions make reliability of operation and plant capacity imperative, it is the belief of the author that many of these plants could utilize relatively cheap, poor grades of fuel with an assurance of both reliability and capacity and a net financial gain. The most difficult problem seems to be that of procuring thoroughly competent men for the careful supervision of such installations. The tabulated figures following, showing the results of Bureau of Mines tests, indicate the possibilities of using in gas producers the fuels represented.

Results of fuel tests, showing adaptability of certain fuels for use in gas producers.

Source of fuel.	Variety or size of fuel.	Ash.	Moisture.	Quantity of fuel, as fired, consumed in producer per brake horsepower-hour.
		Per cent.	*Per cent.*	*Pounds.*
New Mexico	Run of mine	19.63	3.62	1.10
Tennessee	do	20.57	3.55	1.45
Iowa		20.70	16.69	1.56
Wyoming		20.72	9.44	1.70
Do	Run of mine	21.73	8.65	1.83
Illinois	Bone	23.12	8.67	2.88
Brazil, South America	Run of mine	23.44	10.96	2.02
West Virginia	Bone	28.08	2.91	1.26
Pennsylvania	Washery refuse	30.35	2.68	2.34
Do	do	31.89	2.25	2.76
West Virginia	Bone	43.74	.47	1.65

If these figures are compared with the results on page 25, it will readily be seen that even after due allowance has been made for the difference between the operation of a testing station and that of the average commercial plant, the possibilities from these high-ash fuels warrant thoughtful consideration by the progressive managers of commercial installations.

The manager of a large steel plant in England realizes that his ability to maintain the high-grade qualities of the steel, for which his company has been so long noted, and at the same time to reduce the cost of manufacture to a point below that of his competitors depends on a reduction in the cost of generating power. He is, therefore, seriously attacking the immense refuse heaps that have been accumulating for years at the colliery connected with the steel plant. The material from these refuse heaps is passed through a crusher and then to a washer where approximately one-half of the nonconbustible material is removed. The washed fuel, containing approximately 25 per cent of noncombustible material is then utilized in rotary eccentric-grate producers, to the entire satisfaction of the company and with a reduction in the cost of the product of the plant.

In sharp contrast is a plant of several thousand horsepower previously mentioned. This plant is operated in the interests of a large corporation in the United States, which owns extensive bituminous-coal mines not distant from the plant. The operating company sends its own bituminous coal to the market and uses Pennsylvania anthracite at a cost of over $11 per ton in its producer-gas plant. Owing to the lower labor charge when anthracite is used, the records of the plant show, for periods of a year or more, that the cost of operation with bituminous coal, if charged on the books at $8 per ton, is considerably more than with anthracite coal. Although at the prices indicated, the anthracite fuel seems to give the better results, it is a question of no little importance as to whether refuse material from the company's mines might not, even with higher labor costs, prove the least expensive fuel, especially as the plant has ample spare producer capacity.

DATA ON FUEL USED AT COMMERCIAL PLANTS.

The Bureau of Mines sent form letters to numerous producer-gas plants, inquiring as to the character of fuel used, the reasons, if any, for having changed fuels, the reasons why certain plants did not use local fuels, and the necessity for a low sulphur content in the coal. The responses may be briefly summarized as follows, the numbers being those assigned to the replies as received. The figures for fixed carbon, volatile matter, moisture, sulphur, and ash are percentages:

Summary of replies to queries regarding operation of producer-gas plants.

UP-DRAFT PLANTS.

Plants using anthracite.

1. Fuel, buckwheat. Low-sulphur coal necessary, as high-sulphur coal causes back-firing.

9. Fuel, buckwheat. Pea coal has been used. Buckwheat is fully as cheap and works nearly as well. Low-sulphur coal not necessary for successful operation.

16. Fuel, buckwheat, from ⅜-inch down. Analysis follows:

Fixed carbon	82.47
Volatile matter	5.29
Moisture	.....
Ash	12.29

This plant formerly used gas-house coke, but the anthracite works with greater ease, as the coke clinkered badly and gave trouble from tar in the engines.

19. Fuel, Pennsylvania No. 1 buckwheat. Low-sulphur coal regarded as necessary to prevent clinkering.

20. One 200-horsepower producer uses pea anthracite, and one 250-horsepower producer uses run-of-mine bituminous coal. The pea anthracite is used in the spare producer, the other producer having been modified for the use of coke, which is a by-product from the gas department.

36. Fuel, No. 1 buckwheat. Pea anthracite formerly used. Buckwheat costs less and seems to give about the same results.

Plants using bituminous coal.

3. Fuel, Pocohontas slack. Analysis follows:

Fixed carbon	73.60
Volatile matter	17.70
Moisture	1.45
Ash	7.25
Sulphur (separately determined)	.80
British thermal units in coal, as fired	14,800

Formerly used Alabama coal. Unsatisfactory. Too much tar. Caused preignition and variable gas quality. Peat is mined in the vicinity. Was tried, mixed with coal, and did fairly well, but gave an offensive, sickening gas. No ill effects from sulphur in coal, except possibly in causing coal-pile fires.

5. Fuel, bituminous coal from Puritan mines, Cambridge, Ohio, ¾-inch lump. Have experimented with other coals, but the above meets requirements best. Other coal is mined in the vicinity, but it is too high in sulphur. Low-sulphur coal is required, as the sulphur fumes cause distress.

12. Fuel, No. 2 washed nut bituminous coal that passes over a ¾-inch and through a 2½-inch screen. The proximate analysis is:

Fixed carbon	54.83
Volatile matter	29.91
Moisture	7.90
Ash	7.36
Sulphur (separately determined)	.48
British thermal units in coal as fired	12,296

Low-sulphur coal is regarded as essential in the operation of this plant to prevent sulphur fumes throughout the factory and a sulphur coating on the manufactured product as the gas is used for annealing.

17. Fuel, Hocking Valley coal in nut sizes passing over 1-inch and through 2-inch screens. The two grades used show—

Fixed carbon	51.67	57.60
Volatile matter	38.26	34.20
Moisture	9.24	4.99
Ash	10.07	3.21
Sulphur (separately determined)	3.42	.73
British thermal units in coal as fired	12,522	13,658

The second fuel is reported to give not enough gas and to be too high in by-products.

21. Owing to the fact that the gas is used in connection with furnaces and the products of combustion are discharged into the room, considerable irritation of the lungs and throat is experienced, which seems to cause ill health among the employees unless the percentage of sulphur in the fuel is kept low. The fuel used contains less than 1 per cent sulphur.

25. Fuel, Hocking Valley bituminous nut coal. The analysis follows:

Fixed carbon	47.01
Volatile matter	36.67
Moisture	9.30
Ash	6.97
Sulphur (separately determined)	1.98

The company used anthracite for the first two years, but on the addition of new producers changed to bituminous coal, with a saving due to the difference in price. They find that the present plant operates more easily on bituminous coal than the former producers did on anthracite.

33. The analysis of the fuel used follows:

Fixed carbon	69.2
Volatile matter	21.3
Moisture	2.5
Ash	7.0
Sulphur (separately determined)	1.5
British thermal units in coal on dry basis	14,760

37. Fuel, bituminous, Westmoreland (Pa.) run-of-mine coal, showing 14,200 British thermal units on the dry basis. Anthracite coal is mined within 50 miles, but the bituminous coal works more freely and is less expensive.

39. Fuel, New River and Pocahontas (W. Va.) coal reduced to extreme fineness before received. Typical analysis of the New River coal follows:

Fixed carbon	73.6
Volatile matter	20.4
Moisture	1.04
Ash	4.96
Sulphur (separately determined)	.91
British thermal units in coal as fired (approximately)	14,500

Considerable carbon in ash at times because of uneven burning of fuel bed. Have used Pocahontas. No difference noted in general operation between Pocahontas and New River. Some wood is available in the vicinity, but the high cost of procuring it and the uncertainty as to successful gasification in the producers installed prevent its use. No effects, ill or otherwise, have been noted from sulphur.

Plants using lignite.

4. Fuel, Texas, screened, large-lump lignite. Analysis of coal as received:

Fixed carbon	27.02
Volatile matter	36.92
Moisture	29.20
Ash	6.86
Sulphur (separately determined)	.58
British thermal units in coal as received	7,442

Obtain better gas with coal of low-sulphur content.

11. Fuel, lignite screenings from two mines, showing a proximate analysis as follows:

	Mine 1.	Mine 2.
Fixed carbon	17.50	12.13
Volatile matter	47.55	48.18
Moisture	27.21	31.81
Ash	8.21	7.88
Sulphur (separately determined)	.44	1.03
British thermal units in coal as fired	7,073	5,561
British thermal units in coal on dry basis	9,717	8,153
British thermal units in coal combustible	10,952	9,217

Formerly this plant used lignite lump, but the cheaper grade gives satisfactory results. This cheaper fuel is regarded by the mine operators as of lower grade than the lignite lump. It is lower in heat value and is also of a grade not usually marketable. It works with slightly less ease than the lump lignite, but as it costs less and is really worked with little difficulty it is regarded as a very satisfactory fuel. This lignite is mined in the vicinity of the plant. Low-sulphur fuel is not regarded as essential for successful operation.

38. Fuel, lignite, pea-screenings, near Centralia, Wash. Several other Washington lignites have been tried, but all cost more and were no better or not so good on account of hotter fire and more clinker. The present fuel is of lower grade and highest in ash but works better.

Plants using wood.

13. Fuel, waste hardwood in lengths of 1 foot or less, and about 1 to 3 inches in diameter, together with some bark and sawdust. This hardwood runs about 35 to 60 per cent moisture as used. During the early operation of the plant sticks 1½ to 3 feet long were used, but some difficulty was experienced with arching of the fuel bed and the formation of pockets that burned out the gas.

26. Fuel, cypress refuse from the hog machine. Brought about 1 mile from the mill. Chips and sawdust also used. The wood as fired yielded 5,400 British thermal units, and on dry basis 10,000 British thermal units. Other fuels tried in this plant are: Anthracite, bituminous coal, coke breeze, and pine hog. Coal or coke was found inferior to the wood refuse, as the cypress hog gives a more constant quality of good gas. It works with greater ease and costs less than the other fuels. Low sulphur content is regarded as important because of the deleterious action of H_2SO_4 on the producer plant and engine.

DOWN-DRAFT PLANTS.

Plants using bituminous coal.

8. Fuel, Big Muddy (Ill.) run-of-mine coal. Analysis follows:

Fixed carbon	50.92
Volatile matter	32.80
Moisture	9.40
Ash	6.88
Sulphur (separately determined)	1.56
British thermal units in dry coal	13,553

Satisfactory in price and results. Is regarded as high grade. Makes good gas with little clinker. No trouble has been experienced on account of sulphur.

20. One 200-horsepower producer uses pea anthracite and one 250-horsepower producer uses run-of-mine bituminous coal with—

Fixed carbon	76.80
Volatile matter	17.11
Moisture	1.39
Ash	4.70
Sulphur (separately determined)	1.01

The pea anthracite is used in the spare producer, the other producer having been modified for the use of coke, which is a by-product from the gas department.

22. Uses Pocahontas slack. Analysis follows:

Fixed carbon	81.41
Volatile matter	13.23
Moisture	.54
Ash	4.82
Sulphur (separately determined)	.50
British thermal units in slack as fired	14,769

Experiments have been made with other fuels, but no other fuel has been used for any length of time. The lower the sulphur the better the results.

23. Fuel, West Virginia bituminous coal run through a 4-inch crusher.

Fixed carbon	53.89
Volatile matter	36.01
Moisture	3.45
Ash	6.65
Sulphur (separately determined)	2.22
British thermal units in coal as fired (average of 49 cars)	13,741

This company has used other West Virginia coals and Indiana coal but finds the present coal gives a larger producer output. It is of higher grade than the other fuels tried, higher in carbon, lower in ash, and works with greater ease. It costs more than the other fuels.

24. Fuel, bituminous southwestern Illinois coal, 1½ to 7 inches. Analysis follows:

Fixed carbon	55.51
Volatile matter	31.78
Moisture	9.02
Ash	3.69
Sulphur (separately determined)	.52
British thermal units in dry coal	12,805

Run-of-mine coal from the same mine and a few other similar Illinois coals have been used. The run-of-mine coal was changed because of too much slack, as a more uniform grade of coal is preferred. The present fuel is regarded as of higher grade than that previously used, although it comes from the same mine. It is freer from dirt and of more suitable size for producer use. It operates with greater ease. It costs more per ton at the mine. Low sulphur is not essential but is preferred.

27. Fuel, Pennsylvania run-of-mine bituminous coal. The average analysis of the coal in four cars follows:

Fixed carbon	76.94
Volatile matter	13.70
Moisture	1.47
Ash	7.89
Sulphur (separately determined)	1.18
British thermal units in coal as fired	14,535

When this particular grade of coal can not be procured a coal yielding 13,365 British thermal units is substituted. The latter fuel is higher in volatile matter, ash, and sulphur, and lower in fixed carbon. It is not necessary to run the fires quite as hot as with the former fuel on account of the higher volatile matter. The second coal costs less than the first. Low sulphur content is regarded as essential. On account of corrosion the 3-inch steel economizer tubes last only about 18 months.

28. Fuel, West Virginia Pocahontas coal, running from dust to 0.75 inch in size. As fired it yields 13,500 to 14,000 British thermal units, and on the dry basis it yields 14,000 to 14,500 British thermal units.

29. Fuel, Virginia bituminous run-of-mine coal and slack. Washed slack has been used, but a change was made to the fuel first mentioned because no more washed slack could be purchased. The present fuel is regarded as of lower grade, although it works better in the producer and costs less.

35. Fuel, Berwyn and White Scalp level, South Fork (Pa.) coal. Analysis:

Fixed carbon	77.0
Volatile matter	17.0
Moisture	.5
Ash	5.5
Sulphur (separately determined)	1.0
British thermal units in coal as fired	14,834

This is a higher-grade coal and costs more than that previously used. The change was made as better results can be obtained with the present coal, as it works with greater ease and does not cake or clinker. Low sulphur content is regarded as necessary in order to eliminate the action of sulphuric acid.

DOUBLE-ZONE PLANTS.

Plants using bituminous coal.

2. Fuel, New Mexico bituminous chestnut coal. Costs less than coal previously used in steam plants. Low-sulphur coal not necessary.

8. Fuel, Big Muddy (Ill.) run-of-mine coal. Analysis follows:

Fixed carbon	50.92
Volatile matter	32.80
Moisture	9.40
Ash	6.88
Sulphur (separately determined)	1.56
British thermal units in coal on dry basis	13,553

Satisfactory in price and economy. It is regarded as high grade. Makes good gas with little clinker. No trouble has been experienced on account of sulphur.

Plants using lignite.

31. Fuel, lignite, size about 3-inch cubes.

Fixed carbon, approximately	40
Volatile matter	34
Moisture	18
Ash	6
British thermal units in coal as fired	11,400

POUNDS OF FUEL PER SQUARE FOOT OF FUEL-BED AREA PER HOUR.

One of the most important commercial items connected with the design and the operation of gas producers is the determination of the number of pounds of fuel consumed per square foot of fuel-bed area per hour. This rate of fuel consumption varies radically with different types of plants and with different grades and different types of fuel and has led to much difficulty in designing and in rating producers. Early work in this country followed European practice almost entirely and thereby occasioned much trouble in connection with the rating of pioneer plants, and brought about the ultimate failure of many of them. Under certain European conditions fuels of a definite grade are specified, so that high rates of fuel consumption may be obtained. It is not impossible to obtain similar rates of consumption under corresponding circumstances in this country, but as selected fuels are seldom obtainable except for special tests, it has been found that in general in the United States the rate of fuel consumption per square foot of fuel-bed area does not average much over one-half the consumption originally guaranteed by early manufacturers. This fact has, of course, led to a decided modification in the design of many plants.

Although the possible rate of burning high-grade fuel may be relatively high, much depends on the method of operation and the character of the fuel as to the actual commercial results obtained. Certain types of fuel lend themselves more readily to high rates of fuel consumption than others, and with a given fuel the rate of burning will vary with the nature of the gas-generating process, the down-draft plant consuming practically twice as much fuel per square foot of fuel-bed area per hour as the up-draft. In 1911 the writer[a] stated that down-draft plants were in operation that consumed more than 40 pounds of lignite per square foot of fuel-bed area per hour, and that up-draft producers could gasify comparatively large quantities of fuel per hour for relatively short periods, but that in actual operation with ordinary grades of fuel it was doubtful whether the consumption in the up-draft plants would exceed 15 to 16 pounds per square foot of fuel-bed area per hour. The normal figure at that time was stated to be much nearer 10 pounds, and it was said that 10 pounds would prove to be a high figure for fuels having a large percentage of ash or a sulphur content that tended to produce serious clinkering. It is interesting to compare the figures mentioned with the figures from several plants in commercial operation at the present time, as furnished by the owners or operators of the plants. Such figures are presented in the pages following.

FUEL CONSUMPTION OF UP-DRAFT PLANTS USING ANTHRACITE COAL.

The fuel-consumption figures for certain plants using anthracite coal follow:

Fuel consumption of up-draft gas producers using anthracite coal.

Plant No.	Horsepower of each gas generator.	Area of fuel bed.	Fuel as fired per square foot of fuel-bed area per hour.	
			As rated.	As actually operated.
		Square feet.	*Pounds.*	*Pounds.*
14	75	7.1	18.0	....
9	160	12.6	16.0	12.7
34	250	38.5	7.8	....
18	300	42.5	8.7	....
19	300	44.2	6.8	7.3
36	300	44.2	8.5	4.5
16	400	56.6	9.0	....
Average	....	....	10.7	....

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[a] Fernald, R. H., and Smith, C. D., Résumé of producer-gas investigations: Bull. 13, Bureau of Mines, 1911, p. 45.

The average consumption of the seven plants listed, on the basis of the manufacturers' rating, was 10.7 pounds per square foot of fuel-bed area. The number of plants reporting operating figures for anthracite coal is not enough to permit the presentation of averages, although it is probable that the average figure for commercial operation for up-draft plants using anthracite as a fuel is not far from the average given on the basis of the manufacturers' rating.

FUEL CONSUMPTION OF UP-DRAFT PLANTS USING BITUMINOUS COAL.

Figures covering fuel consumption of gas producers using bituminous coal follow:

Fuel consumption of up-draft gas producers using bituminous coal.

Plant No.	Horsepower of each gas generator.	Area of fuel bed.	Fuel as fired per square foot of fuel-bed area per hour.	
			As rated.	As actually operated.
		Square feet.	*Pounds.*	*Pounds.*
37	200	23.7	7.0	7.0
3	200	25.0	10.0	7.6
33	250	28.3	10.6	7.6
17	250	33.1	10.0	13.7
12	200 300	24.0 42.0	10.4 9.5	7.4
25	300 400	40.0 50.0	9.1 8.0	8.5
39	370	50.2	10.0	6.1
21	650 1,000	50.0 78.0	....	8.7
5	1,000	70.9	7.0	7.1
12	2,500	210.0	14.3	13.1
Average	....	....	10.4	8.7

For the up-draft plants operating on bituminous coal the range of actual operating rates of fuel consumption seems to be from approximately 6 to more than 13.5 pounds per square foot of fuel-bed area per hour. This marked difference might, on first thought, be supposed to be due to differences in the character of the fuels, but an examination of the records submitted shows that actually the higher-grade fuel is used in the plant reporting the low rate of fuel consumption per square foot of fuel-bed area per hour.

The coal used in the plant reporting the 6-pound rate averaged 14,500 British thermal units as fired and contained 5 per cent ash and 0.9 per cent sulphur, whereas the coal used in the plant reporting the 13.7-pound rate averaged 12,520 British thermal units and contained 10.1 per cent ash and 3.4 per cent sulphur, and the coal in the plant reporting the 13.1-pound rate averaged 12,300 British thermal units and contained 7.4 per cent ash and 0.5 per cent sulphur.

The inference is that the plant with the 6-pound rate was operated much below the capacity of the producer.

Although there seems to be little direct relation between the rated fuel consumption for the individual plants and the number of pounds of fuel actually burned per square foot of fuel-bed area, the average figures check within reasonable limits and indicate that approximately 8.5 pounds per square foot of fuel-bed area per hour with good-grade bituminous coal is a reasonable figure for an up-draft producer gas plant.

FUEL CONSUMPTION OF UP-DRAFT PLANTS USING LIGNITE.

The figures for fuel consumption in gas producers using lignite follow:

Fuel consumption of up-draft gas producers using lignite.

Plant No.	Horsepower of each gas generator.	Area of fuel bed.	Fuel as fired per square foot of fuel-bed area per hour.	
			As rated.	As actually operated.
		Square feet.	*Pounds.*	*Pounds.*
4	100	19.6	11.5	11.5
7	100	20.4	....	8.6
11	250	38.5	....	14.8
38	300	41.3	10.0	8.5
Average	....	....	10.8	10.8

The returns cited for lignite in up-draft plants indicate that approximately 11 pounds of lignite is burned per square foot of fuel-bed area per hour in plants of this type.

Tests at the Government testing station at St. Louis in 1904–1906 indicated that a higher average rate may easily be obtained with lignite in up-draft plants.

FUEL CONSUMPTION OF UP-DRAFT PLANTS USING WOOD.

Figures for fuel consumption in gas producers using wood follow:

Fuel consumption of up-draft gas producers using wood.

Plant No.	Horsepower of each gas generator.	Area of fuel bed.	Fuel as fired per square foot of fuel-bed area per hour.	
			As rated.	As actually operated.
		Square feet.	*Pounds.*	*Pounds.*
13	150	50.3	12.0	....
26	200 280	33.3 50.3	14.0	14.5

FUEL CONSUMPTION OF DOWN-DRAFT PLANTS USING BITUMINOUS COAL.

Figures showing fuel consumption in certain plants using bituminous coal follow:

Fuel consumption of down-draft gas producers using bituminous coal.

Plant No.	Horsepower of each gas generator.	Area of fuel bed.	Fuel as fired per square foot of fuel-bed area per hour.	
			As rated.	As actually operated.
		Square feet.	*Pounds.*	*Pounds.*
22	250	17.4	14.4	14.4
27	250	17.4	17.0	…………
35	250	20.4	14.7	14.7
28	375	38.5	13.0	…………
28	500	50.0	12.5	…………
8	800	36.0	33.4	18.4
23	1,500	60.0	27.5	23.5
24	1,500	60.0	20.0	16.7
Average	…………	…………	19.1	17.5

For the down-draft plants of the double-generator type the figures reported are perhaps not sufficient to warrant conclusive deductions, but when compared with data from other sources they make apparent that the average fuel consumption per square foot of fuel-bed area per hour is nearly correct.

FUEL CONSUMPTION IN DOWN-DRAFT GAS PRODUCER USING LIGNITE.

Figures procured a few years since from a plant using lignite in 1,100-horsepower units of the down-draft double-generator type indicated a normal fuel rate of 33 pounds per square foot of fuel area per hour for 16 hours a day, and the unusual rate of 48 pounds for 8 hours a day. This instance, however, is too exceptional to be regarded as representative of general practice.

FUEL CONSUMPTION OF DOUBLE-ZONE PLANTS USING BITUMINOUS COAL.

Figures showing fuel consumption of double-zone gas producers using bituminous coal follow:

Fuel consumption of double-zone gas producers using bituminous coal.

Plant No.	Horsepower of each gas generator.	Area of fuel bed.	Fuel as fired per square foot of fuel-bed area per hour.	
			As rated.	As actually operated.
		Square feet.	*Pounds.*	*Pounds.*
2	250	29.3	24.0	10.3
8	500	47.0	17.0	…………

As operating returns were received for only one double-zone plant, no definite direct deductions can be made, but a review of the results of tests of such a plant indicates the rate of fuel consumption under test conditions to be not far from 15 pounds per square foot of fuel-bed area per hour with bituminous coal and 23 pounds with lignite.

CONCLUSIONS REGARDING COAL CONSUMPTION.

A study of the operating reports submitted indicates that the average rate of fuel consumption in producer-gas plants is considerably higher than a few years ago. This betterment is due, perhaps, to two reasons—first, a more efficient operation of the plants themselves, and, second, an increasing tendency to use higher grade fuel. The first result is to be commended, but the second should be deplored.

Although too much stress must not be laid on the following values, the deductions from the operating figures supplied above, coupled with personal observation of many other plants, seem to indicate the following approximate values for fuel consumption per square foot of fuel-bed area per hour as representative of good general commercial practice.

Figures for fuel consumption in gas producers representing good general practice.

Kind of plant.	Fuel consumption per square foot of fuel-bed area per hour with—							
	Anthracite coal.		Bituminous coal.		Lignite.		Peat.	Wood.
	Average.	Maximum.	Average.	Maximum.	Average.	Maximum.	Average.	Average.
Up-draft using—	*Pounds.*	*Pounds.*	*Pounds.*	*Pounds.*	*Pounds.*	*Pounds.*	*Pounds.*	*Pounds.*
Fuel as fired	10.0	14.0	8.5	14.0	12.0	17.0	15.0	14.0
Fuel dry	10.0	13.5	8.0	13.0	8.5	12.0	12.0	
Down-draft using—								
Fuel as fired			17.5	23.5	26.5	31.5	35.5	
Fuel dry			16.5	22.0	18.5	22.0	25.5	
Double-zone using—								
Fuel as fired			13.5	18.5	21.5	27.0		
Fuel dry			12.5	17.5	15.0	19.0		

POUNDS OF FUEL PER HORSEPOWER-HOUR.

Producer-gas investigations of the United States Geological Survey and of the Bureau of Mines, conducted with plants not above the average in efficiency, showed the following approximate fuel consumption per brake horsepower per hour:

Results of producer-gas investigations, showing fuel consumption per brake horsepower per hour with different fuels.

Condition of fuel.	Fuel consumption per brake horsepower-hour with—						
	Bituminous coal.			Lignite.			Peat.[a]
	Average.	Maximum.	Minimum.	Average.	Maximum.	Minimum.	Average.
	Pounds.	*Pounds.*	*Pounds.*	*Pounds.*	*Pounds.*	*Pounds.*	*Pounds.*
Fired	1.3	2.0	0.8	2.0	2.8	1.5	2.6
Dry	1.2	1.8	.8	1.63	2.02	1.35	2.0

[a] One sample only of peat.

Although these figures were procured during the progress of regular tests, the conditions outlined in previous reports of the Bureau of Mines indicate clearly that equally good results should be readily obtained in the average commercial producer-gas plant. With this in mind, the following results, reported by the operators and owners of several plants, are of peculiar interest:

Results of operation of producer-gas plants showing fuel consumed per brake horsepower-hour with different fuels.

Plant No.	Horsepower of each gas generator.	Total horsepower of plant.	Heat value of fuel as fired.	Fuel fired per brake horsepower-hour.	
				Rating.	Operating.
ANTHRACITE COAL.					
			B. t. u.	*Pounds.*	*Pounds.*
14	75	150		1.67	
19	300	300		1.00	1.30
36	300	300		1.25	1.30
16	400	400		1.25	
18	300	600		1.23	
9	160	640		1.25	1.50
34	250	1,500		1.20	1.26
Average				1.26	1.34
BITUMINOUS COAL.					
29	200	200		1.10	1.00
20	250	250		1.25	
3	200	400	14,800	1.25	2.40
37	200	400	14,000		1.65
17	250	500	12,520	1.30	
33	250	500	14,370	1.20	1.00
27	250	500	14,535	1.10	
22	500	500	14,770	1.00	1.20
12	200 300	1,000	12,300	1.30	1.40
35	250	1,000	14,835	1.20	1.00
39	370	1,100	14,500	1.37	1.80
25	300 400	1,400		1.10	1.40
2	250	1,500		2.80	2.00
28	375 500	1,750	13,750	1.29	1.00
8	500 800	4,200	12,270	1.60	1.20
23	1,500	6,000	13,740	1.10	1.15
24	1,500	9,000	11,540	.80	
Average				1.30	1.40
LIGNITE.					
4	100	100	7,440	2.25	2.50
7	100	100			2.40
38	300	300			2.00
31	200	400	11,400	1.50	3.00
Average					2.48
WOOD.					
13	150	150		4.00	
26	200 280	1,040	5,400	2.50	3.33

A more direct comparison between the results of commercially operated plants and those obtained at the Government testing station may be had by an inspection of the following tabulation:

Comparative results of operating producer-gas plants by the Government and by commercial companies.

Operator.	Fuel consumed per brake horsepower-hour with—										
	Anthracite.			Bituminous coal.			Lignite.			Peat.[a]	Wood[a]
	Average.	Maximum.	Minimum.	Average.	Maximum.	Minimum.	Average.	Maximum.	Minimum.	Average.	Average.
	Lbs.	*Lbs.*	*Lbs.*	*Lbs.*	*Lbs.*	*Lbs.*	*Lbs.*	*Lbs.*	*Lbs.*	*Lbs.*	*Lbs.*
Bureau of Mines	....	....	....	1.3	2.0	0.8	2.0	2.8	1.5	2.6	....
Commercial plants	1.3	1.5	1.3	1.4	2.4	1.0	2.5	3.0	2.0	....	3.3

[a] One sample only used.

Figures showing the heat value of the fuel and the actual consumption of fuel as fired per horsepower-hour for different plants follow:

Heat value of fuel used at different gas producers and fuel consumption per horsepower-hour.

Plant No.	Heat value of fuel.	Fuel fired per horsepower-hour.	Plant No.	Heat value of fuel.	Fuel fired per horsepower-hour.
	B. t. u.	*Pounds.*		*B. t. u.*	*Pounds.*
4	7,440	2.50	37	14,000	1.65
31	11,400	3.00	33	14,370	1.00
8	12,270	1.20	22	14,770	1.20
12	12,300	1.40	3	14,800	2.40
23	13,740	1.15	35	14,835	1.00
28	13,750	1.00			

It is at once evident that there is no direct relation between the independent results obtained by different operators and the heat value of the fuel used. This lack of relationship is to be expected, as individual plant conditions are so radically different and producer-gas power is relatively so new that some plants are necessarily much more efficiently operated than others, a fact convincingly borne out by a personal inspection of a large number of plants. Even plants owned by the same corporation, situated in the same district and using the same general grades of fuel, may bear little relation to each other in the results obtained. In one plant after the power used had been changed from steam to producer gas the cost per ton of finished product was reduced to less than one-half of the former cost, whereas in another plant controlled by the same company the cost per ton of finished product was practically doubled after a similar introduction of producer gas. The difference in the two results seems to have been due to the difference in the methods of the superintendents in charge. One superintendent was a live, wide-awake man who believed it essential to have his power plant kept in perfect condition for the successful manufacture of his product. As a result, he has never shut down for want of power.

The other superintendent felt that the power plant ought to take care of itself. The appearance of the plant testified plainly to his expressed views and serious shutdowns were frequent.

DEPTH OF FUEL BED.

Data supplied by various producer-gas companies regarding the depth of fuel bed used follow:

Data regarding depth of fuel bed used in various producer-gas plants.

UP-DRAFT PLANTS.

PLANTS BURNING ANTHRACITE COAL.

Plant No.	Grate area.	Approximate quantity of fuel burned per hour.	Depth of bed.
	Square feet.	*Pounds.*	*Feet.*
14	7.1	125	4
9	12.6	160	2.5
6	13.6	......	3
36	44.2	200	6
32	38.5	......	2.75
34	38.5	300	2
19	44.2	300	10
16	56.6	500	4

PLANTS BURNING BITUMINOUS COAL.

37	23.7	165	2
3	25.0	190	3
39	50.2	300	1.5
25	40.0	340	2.5
25	50.0	425	2.5
21	50.0	435	1.75
17	33.1	450	4
5	70.9	500	3
21	78.0	680	1.75
12	210.0	2,750	6.5

PLANTS BURNING LIGNITE.

15	......	......	4
4	19.6	225	2
38	41.3	350	6.5
11	38.5	570	1.5

PLANTS BURNING WOOD.

13	50.3	605	8
26	50.3	725	7

Data regarding depth of fuel bed used in various producer-gas plants—Continued.

DOWN-DRAFT PLANTS.

PLANTS BURNING BITUMINOUS COAL.

Plant No.	Grate area.	Approximate quantity of fuel burned per hour.	Depth of bed.
	Square feet.	*Pounds.*	*Feet.*
29	....	220	5
22	17.4	250	5
27	17.4	295	6
35	20.4	300	12
28	50.0	625	6
8	36.0	1,200	8.5
23	60.0	1,400	9

DOUBLE-ZONE PLANTS.

PLANT BURNING BITUMINOUS COAL.

2	29.3	310	12

PLANT BURNING LIGNITE.

31	....	....	8

COMPOSITION OF GAS.

The composition of producer gas varies with the type of producer, the methods and skill used in operating it, the uniformity and regulation of the air and steam supply, the kind and quality of fuel used, the depth of fuel bed, the distribution of the fuel, and the uniformity in size of the fuel. The variations in the composition of producer gas under different operating conditions are readily shown by the following results of analyses of the gas produced in different plants.

Results of analyses of gas from different types of gas producers.

[Results represent per cent by volume.]

UP-DRAFT PLANTS.

PLANTS BURNING ANTHRACITE COAL.

Plant No.	CO	CH_4	C_2H_4	H_2	CO_2	O_2	N_2
14	24.0	....	....	12.0	3.5	0.2	60.3
19	21.4	....	....	19.0	7.5	.3	51.8
Average	22.7	....	....	15.5	5.5	.3	56.0

PLANTS BURNING BITUMINOUS COAL.

37	27.5	2.2	0.7	6.5	2.9	0.2	60.0
17	25.0	4.3	.0	14.6	5.2	.9	50.0
39	21.8	2.8	.0	11.3	6.4	.4	57.3
25	23.6	4.4	.0	14.2	4.9	1.3	51.6
21	24.0	4.6	.0	11.6	4.6	.0	55.2
Average	24.4	3.7	.1	11.6	4.8	.6	54.8

Results of analyses of gas from different types of gas producers—Continued.

UP-DRAFT PLANTS—Continued.

PLANTS BURNING LIGNITE.

Plant No.	CO	CH_4	C_2H_4	H_2	CO_2	O_2	N_2
38	16.2	5.0	0.0	25.0	6.9	1.3	45.4
11	[a] 21.0				6.6	2.5	
11	[b] 9.8				16.6	3.2	

PLANT BURNING WOOD.

26	13.6	8.0	0.0	4.0	12.9	0.0	61.7

DOWN-DRAFT PLANTS.

PLANTS BURNING BITUMINOUS COAL.

35	24.0	1.6	0.0	14.3	2.0	0.2	57.9
8	21.2	3.0	.0	9.3	5.0	.3	61.2
24	25.6	.0	1.0	13.6	5.0	.0	54.8
Average	23.6	1.5	.3	12.4	4.0	.2	58.0

DOUBLE-ZONE PLANTS.

PLANT BURNING BITUMINOUS COAL.

8	23.2	2.8	0.0	11.0	6.2	1.1	55.7

PLANT BURNING LIGNITE.

31	10.4	3.8	0.0	17.2	15.4	0.4	52.8

OIL-GAS PLANTS.

PLANTS BURNING CRUDE OIL.

Plant No.	CO	CH_4	H_2S	C_xH_y	H_2	CO_2	O_2	N_2
10	10.2	6.1	0.0	3.8	10.6	6.1	0.0	63.2
30	7.4	12.7	3.1	2.6		4.5		69.3

[a] No steam.

[b] With steam.

The averages of several typical analyses of producer gas from the Bureau of Mines testing plant and the averages of the figures presented for plants in commercial operation are given below. Attention is called to the fact that the analyses may not be strictly comparable, because methods of analyses may have differed slightly.

Average results of analyses of producer gas from Bureau of Mines testing plants and from commercial plants.

[Results represent per cent by volume.]

UP-DRAFT PLANTS.

Constituent.	Gas from anthracite coal.		Gas from bituminous coal.		Gas from lignite.		Gas from peat.[a]		Gas from wood.[a]	
	Bureau of Mines.	Commercial plants.	Bureau of Mines.	Commercial plants.	Bureau of Mines.	Commercial plants.	Bureau of Mines.	Commercial plants.	Bureau of Mines.	Commercial plants.
Carbon monoxide (CO)	....	22.7	18.28	24.4	21.00	....	21.0	....	....	13.6
Methane (CH_4)	....	.0	3.12	3.7	2.20	....	2.2	....	....	8.0
Ethylene (C_2H_4)	....	.0	.18	.1	.40	....	.4	....	....	.0
Hydrogen (H_2)	....	15.5	12.90	11.6	18.50	....	18.5	....	....	4.0
Carbon dioxide (CO_2)	....	5.5	9.84	4.8	12.40	....	12.4	....	....	12.9
Oxygen (O_2)	....	.3	.04	.6	.00	....	.0	....	....	.0
Nitrogen (N_2)	....	56.0	55.64	54.8	45.50	....	45.5	....	....	61.7

[a] One sample only.

DOWN-DRAFT PLANTS.

Constituent.	Gas from anthracite coal.		Gas from bituminous coal.		Gas from lignite.		Gas from peat.		Gas from wood.	
	Bureau of Mines.	Commercial plants.	Bureau of Mines.	Commercial plants.	Bureau of Mines.	Commercial plants.	Bureau of Mines.	Commercial plants.	Bureau of Mines.	Commercial plants.
Carbon monoxide (CO)	....	....	19.1	23.6	15.0	....	14.8	....	....	....
Methane (CH_4)	....	....	1.1	1.5	1.7	....	1.5	....	....	....
Ethylene (C_2H_4)	....	....	.0	.3	.0	....	.1	....	....	....
Hydrogen (H_2)	....	....	11.4	12.4	13.3	....	13.3	....	....	....
Carbon dioxide (CO_2)	....	....	7.6	4.0	11.5	....	12.9	....	....	....
Oxygen (O_2)	....	....	.2	.2	.2	....	.6	....	....	....
Nitrogen (N_2)	....	....	60.6	58.0	58.3	....	56.8	....	....	....

HEAT VALUE OF GAS.

Figures showing the heat value of the gas from different fuels determined from the average of a large number of tests reported by the Bureau of Mines and also from the figures submitted by the operators of plants in commercial operation are presented below:

Average heat values of gas from different fuels when used in various types of gas producers.

[Figures represent British thermal units per cubic foot of gas.]

UP-DRAFT PLANTS.

Location of gas producer.	Gas from anthracite coal.		Gas from bituminous coal.		Gas from lignite.		Gas from peat.		Gas from wood.	
	Average.	Maximum.	Average.	Maximum.	Average.	Maximum.	Average.	Maximum.	Average.	Maximum.
Bureau of mines testing station	....	....	152	176	158	188	[a] 175	....	....	....
Commercial plants	138	....	151	175	157	185	....	....	[a] 133	....

[a] One sample only.

Average heat values of gas from different fuels when used in various types of gas producers—Continued.

DOWN-DRAFT PLANTS.

Location of gas producer.	Gas from anthracite coal.		Gas from bituminous coal.		Gas from lignite.		Gas from peat.		Gas from wood.	
	Average.	Maximum.	Average.	Maximum.	Average.	Maximum.	Average.	Maximum.	Average.	Maximum.
Bureau of mines testing station			110	123	111	127	[a] 115	[a] 119		
Commercial plants			123	130						

DOUBLE-ZONE PLANTS.

Bureau of mines testing station										
Commercial plants			([b])		118					

OIL-GAS PLANTS.

Bureau of mines testing station										
Commercial plants				[c] 215	[c] 230					

[a] Two samples only.
[b] Tests indicate this figure to be approximately 115.
[c] Figures represent two plants burning crude oil.

CUBIC FEET OF GAS PER POUND OF FUEL.

Tabulated data showing the quantity of producer gas available from various fuels used in the different types of plants follow:

Average quantities of gas produced from different fuels in different types of gas producers.

[Figures represent cubic feet of standard (60° F. and 30 inches of mercury) gas per pound of fuel.]

UP-DRAFT PLANTS.

Location of gas producer.	Gas from bituminous coal.				Gas from lignite.				Gas from peat.			
	As fired.		Dry.		As fired.		Dry.		As fired.		Dry.	
	Average.	Maximum.	Average.	Maximum.	Average.	Maximum.	Average.	Maximum.	Average.	Maximum.	Average.	Maximum.
Bureau of mines testing station	61	101	65	104	36	46	46	53	[a] 30		[a] 38	
Commercial plants	75	96										

DOWN-DRAFT PLANTS.

Bureau of mines testing station	65	80	68	82	36	44	52	61	[b] 29	[b] 31	[b] 40	[b] 44
Commercial plants	79	82										

[a] One sample only.
[b] Two samples only.

TIME BETWEEN FUEL CHARGES AND WEIGHT OF FUEL CHARGES.

Figures showing the time between fuel charges and the weight of fuel charges in the various types of plants follow:

Time between fuel charges and weight of fuel charges in different types of gas producers.

UP-DRAFT PLANTS.

PLANTS BURNING ANTHRACITE COAL.

Plant No.	Horsepower of each gas generator.	Average fuel burned per hour.	Length of service per day.	Time between fuel charges.	Weight of fuel charge.
		Pounds.	*Hours.*	*Hours.*	*Pounds.*
6	150	....	24	1 to 6	200
9	160	160	24	.5	200 to 600
32	250	....	3 to 7	2	400
18	300	190	11	24	1,800 to 2,400
19	300	300	24	6	1,000 to 1,500
36	300	200	10	10	2,000
1	350	....	5 to 24	.5	175

PLANTS BURNING BITUMINOUS COAL.

3	200	190	24	1	165
37	200	165	10	.5	80
17	250	450	....	4	1,500
33	250	215	14	.75	250
39	370	300	24	.17	100
21	650 1,000	435 680	10	.13	100
12	2,500	2,750	24	2.5	7,000

PLANTS BURNING LIGNITE.

4	100	225	8	2	500 to 800
7	100	175	11	1	175
15	100	....	14	2 to 3	200 to 300
11	250	570	24	.33	190
38	300	350	24	2 to 3	900

PLANT BURNING WOOD.

26	200 280	....	24	0.5	280

DOWN-DRAFT PLANTS.

PLANTS BURNING BITUMINOUS COAL.

29	200	....	24	0.5	110
27	250	295	10	.25	50
35	250	300	10	.17	50
28	375 500	625	24	.17	100
8	800	1,200	24	.25	....
23	1,500	1,400	24	.09	....

DOUBLE-ZONE PLANTS.

PLANT BURNING BITUMINOUS COAL.

2	250	310	24	1 to 1.5	700

TIME BETWEEN POKINGS OF FUEL BED.

Tabulated data showing the time between pokings of the fuel bed in the various types of plants are presented below:

Time between pokings of fuel bed in different types of gas producers.

UP-DRAFT PLANTS.

PLANTS BURNING ANTHRACITE COAL.

Plant No.	Area of fuel bed.	Depth of fuel bed.	Approximate amount of fuel burned per hour.	Time between pokings.
	Square feet.	*Feet.*	*Pounds.*	*Hours.*
14	7.1	4	125	1
9	12.6	2.5	160	.75
6	13.6	3	...	3 to 6
36	44.2	6	200	10
34	38.5	2	300	1
19	44.2	10	300	8
1	...	...	...	1
16	56.6	4	500	6

PLANTS BURNING BITUMINOUS COAL.

37	23.7	2	165	10
3	25.0	3	190	.33
25	40.0	2.5	340	4
21	50.0	1.75	435	.75
17	33.1	4	450	1
33	28.3	...	215	2
5	70.9	3	500	1
21	78.0	1.75	680	.75
12	210	6.5	2,750	12

PLANTS BURNING LIGNITE.

15	...	4	...	1
7	20.4	...	175	12
4	19.6	2	225	2
11	38.5	1.5	570	12

PLANTS BURNING WOOD.

13	50.3	8	605	0.25
26	50.3	7	725	.16

DOWN-DRAFT PLANTS.

PLANTS BURNING BITUMINOUS COAL.

29	...	5	220	1
22	17.4	5	250	([a])
27	17.4	6	295	.5 to 1
35	20.4	12	300	3
8	36.0	8.5	1,200	([a])
23	60.0	9	1,400	.5

DOUBLE-ZONE PLANTS.

PLANT BURNING BITUMINOUS COAL.

2	29.3	12	310	1

PLANT BURNING LIGNITE.

31	...	8	...	4

[a] Not poked.

TROUBLE FROM HOLES AND CHANNELS IN FUEL.

Data supplied by owners or operators regarding trouble from holes and channels in the fuel bed in the various types of plants are presented below. The numbers preceding each paragraph refer to plant numbers assigned by the author:

Data regarding holes and channels in full bed.

Up-draft Plants.

Plants burning anthracite coal.

1. Causes back-firing trouble.
6. Little trouble from holes and channels.
9. Little trouble, because we use sufficient producer capacity.
14. Not serious. Holes easily closed.
16. Considerable stoking required at times.
19. No trouble.
20. Permit gas to burn in producer.
32. Shut the engine down.
34. Make weak gas.
36. No trouble.

Plants burning bituminous coal.

3. Cause poor gas and clinker.
5. Little trouble.
12. No trouble when producers have proper attention.
17. No trouble.
21. Cause much trouble.
25. No trouble.
33. Not appreciable.
37. Cause weak gas.
39. Serious. Necessitating laborious work to keep the fire even throughout the bed.

Plants burning lignite.

4. Occasional, but never serious.
7. Destroys gas.
11. No trouble. Dropping fuel on fire keeps it down.
38. Very little. Never have this trouble, as little work keeps the fuel bed in fine shape.

Plants burning wood.

13. Permit unfixed oxygen to reach top of fuel bed, causing burning.
26. No trouble.

Down-draft Plants.

Plants burning bituminous coal.

8. No trouble.
20. Permits gas to burn in producer.
22. No trouble.
23. Do not let them occur.
24. Readily noticeable on recording calorimeter. Believed to have affected results seriously.
27. Fire must be closely watched and fuel charged lightly.
28. No trouble.
29. Considerable. At times had to shut plant down.
35. No trouble.

Double-zone Plants.

Plants burning bituminous coal.

2. Quite serious.
8. No trouble.

METHODS OF OVERCOMING VARIATIONS IN QUALITY OF GAS.

Data supplied by owners or operators regarding methods of overcoming variations in the quality of the gas yielded by the various types of gas producers follow:

Data regarding methods used for overcoming variations in gas quality in various types of gas producers.

UP-DRAFT PLANTS.

PLANTS BURNING ANTHRACITE COAL.

Plant No.	Horsepower of each gas generator.	Remarks of owner or operator regarding methods used to overcome variations in quality of gas.
14......	75	None.
6.......	150	Variation of steam feed.
9.......	160	None.
20......	200	Suction producer, none; pressure producer, gasometer.
32......	250	Change setting of mixing valve on engine.
19......	300	Automatic vapor control and proper cleaning methods.
36......	300	Automatically controlling steam supply to producer.
16......	400	Producer automatically regulated.

PLANTS BURNING BITUMINOUS COAL.

3.......	200	Gas holder.
37......	200	By keeping fires regular gas quality varies but very little.
17......	250	None.
33......	260	Do.
39......	370	None, except best possible attention to producer fuel bed.
25......	300, 400	Hand regulation of air temperature admitted to ash bed.
21......	650, 1,000	None, except to reprimand gasmen.
5.......	1,000	Keep bed tight, uniform, and free from air pockets.
12......	200, 300, 2,500	Gas quality checked by recording calorimeter.

PLANTS BURNING LIGNITE.

4.......	100	Change mixture at engine.
7.......	100	Adjustment of levers.
15......	100	More or less free air.
11......	250	Mixing valves on engines. Gas constant except when cleaning fires.
38......	300	None. Little variation.

PLANT BURNING WOOD.

26......	200, 280	None.

DOWN-DRAFT PLANTS.

PLANTS BURNING BITUMINOUS COAL.

29......	200	Close attention to fire.
35......	250	Keep fires and scrubbers clean.
28......	375, 500	20,000-cubic foot gas holder.
22......	500	None, except with fires in generators and mixing in gas holders.
8.......	800	Water gas run to regulate temperature of fires.
23......	1,500	None other than holder.
24......	1,500	Mixture of gas from different producers. Careful watching of fires.

DOUBLE-ZONE PLANTS.

PLANT BURNING BITUMINOUS COAL.

2.......	250	None.

Data regarding methods used for overcoming variations in gas quality in various types of gas producers—Continued.

DOUBLE-ZONE PLANTS—Continued.

PLANT BURNING LIGNITE.

Plant No.	Horsepower of each gas generator.	Remarks of owner or operator regarding methods used to overcome variations in quality of gas.
31......	200	Change mixing valves on engine.

OIL-GAS PLANT.

PLANT BURNING CRUDE OIL.

10......	400	Variations so slight that governor handles them easily.

METHODS OF CLEANING GAS GENERATORS.

The owners and operators of producer-gas plants were asked to describe briefly their method of cleaning the gas generator; they were requested to state the time and the number of men required for cleaning each unit. Brief abstracts of the replies received are tabulated below.

Data regarding methods of cleaning gas generators in various types of producer-gas plants.

UP-DRAFT PLANTS.

PLANTS BURNING ANTHRACITE.

Plant No.	Horse-power of each gas generator.	Total horse-power of plant.	Remarks of owner or operator regarding methods used to clean gas generator.
14......	75	150	Coal and ash pulled through doors above grate; clinkers removed from walls by bar through poke hole; 3 hours' time of 1 man.
6.......	150	450	Half close valve between producer and gas main; remove all ashes between grates and fire with hoe and poker; poke fire down and add fresh fuel; 1 hour's time of 1 man.
9.......	160	640	Draw ash and clinker, allowing fire to settle down to grate; one-half hour's time of 1 man.
32......	250	500	After shutting down at 6 p. m., ashes raked out at water seal; 1 hour's time of 1 man.
19......	300	300	Ash removed from center of fire by rocking grate operated by compressed-air cylinder; fine ash is barred from lining and raked out; 2 hours' time of 1 man.
36......	300	300	Bar lining; shake grate; 1 hour's time of 1 man.
1.......	350	700	Raked out from bottom; 2 to 3 hours' time of 2 men.
16......	400	400	Ash is taken out at two intervals in 24 hours; 1 to 2 hours' time of 1 man.

PLANTS BURNING BITUMINOUS COAL.

3......	200	400	Air and steam cut off for a couple of days; ash and coal removed through water seal; 3 hours' time of 1 man.
37......	200	400	All free ash is removed from bed, leaving fire bridged; bed is then broken down and producer filled; 1½ hours' time of 2 men.
17......	250	500	Shaking the grates and pulling the ashes; three-fourths of an hour's time of 1 man.
39......	370	1,100	Remove sufficient ash from water seal to let bed down to normal level; work loose bed down to new level with poke bars; break clinkers off walls and smash fine with chisel bars and heavy hammers; 12 hours' time of 3 men is the minimum.
25......	300 400	1,400	(*a*) Clean by hand-operated rotary table; (*b*) clean by air-driven shaking grate; 5 minutes' time of 1 man; clean when running.
21......	650, 1,000	3,650	Not much of this to do.
5.......	1,000	4,000	Draw ashes till bed drops 1 foot; poke fire till solid; 1½ hours' time of 1 man.
12......	2,500	2,500	Twenty minutes to 1 hour's time of 1 man.

Data regarding methods of cleaning gas generators in various types of producer-gas plants—Continued.

UP-DRAFT PLANTS—Continued.

PLANTS BURNING LIGNITE.

Plant No.	Horse-power of each gas generator.	Total horse-power of plant.	Remarks of owner or operator regarding methods used to clean gas generator.
4	100	100	Burn low; rake and clean everything out; 6 hours' time of 2 men.
15	100	100	If run 3 days, 24 hours per day, take out everything; if run 14 hours a day, clean every day; for 3 days' run, 3 hours' time of 1 man.
11	250	500	Bar fires, and take out ashes, working around producer several times to prevent fire dropping; if gas becomes weak, stop a while; 1½ hours' time of 2 men.
38	300	300	Run fuel bed down till 3 or 4 feet deep; open hoppers and all poke holes and draw out fire below; fire is kindled by layer of shavings and fine wood on grates about 12 inches deep; cover with layer of 4 inches of good dry coal; after well lighted, fill with coal; close hoppers and poke holes, starting gas washer for draft; 10 hours' time of 2 or 3 men.

PLANTS BURNING WOOD.

13	150	150	Producer cleaned daily by removal of ashes; scrubber pit cleaned every hour or so to avoid tar clogging the main; work done by 1 man.
26	200, 280	1,040	Beds lowered by removing ash once a month through water seal; 3 hours' time of 2 men.

DOWN-DRAFT PLANTS.

PLANTS BURNING BITUMINOUS COAL.

29	200	200	Dig out from bottom and punch down from top; 15 minutes' time of 1 man.
27	250	500	Let producer stand from Saturday till Monday; spray fire with water on top for 15 minutes; let stand for 1 hour; clean everything out; recharge with 2,000 pounds of coke; 10 hours' time of 2 men.
35	250	1,000	Fuel and ash are cleaned out every 2 weeks and new fires started; 4 hours' time of 6 men.
28	375, 500	1,750	Clinker sometimes very hard and must be broken with bar from above; 6 to 10 hours' time of 8 men for each pair of producers.
22	500	500	All ash, refuse, and clinkers removed; 5 hours' time of 3 men.
8	800	3,200	Set is cut out; cooled with water; ashes removed; clinker cut down; 8 hours' time of 4 men.
23	1,500	6,000	Water sprayed through charging door directly after run till fire is cooled; coke withdrawn; ashes follow; 4 men, 3 days, 10 hours each.
24	1,500	9,000	Power-gas producer: Put fire out; remove clinker, coke, and ash; 2 to 4 days' time of 3 to 5 men. Heating-gas producer: Throw fire to one side; clean grate; throw fire back; repeat for other side; 1 to 2 hours' time of 1 man.

DOUBLE-ZONE PLANTS.

PLANTS BURNING BITUMINOUS COAL.

2	250	250	Not necessary to shut down to clean.
8	500	500	See procedure under "Down-draft plants."

OIL-GAS PLANT.

PLANT BURNING CRUDE OIL.

30	....	....	Raise stack valve, shut off blower, remove coke, close door, turn on blast, adjust oil, and lower stack; 10 minutes' time of 1 man.

SERIOUS CLEANING TROUBLES.

Data furnished by owners and operators regarding cleaning troubles in the different types of gas producers are presented in the tabulation following:

Data regarding cleaning troubles in various types of gas producers.

UP-DRAFT PLANTS.

PLANTS BURNING ANTHRACITE COAL.

Plant No.	Horsepower of each gas generator.	Total horsepower of plant.	Remarks of owner or operator regarding cleaning troubles.
14......	75	150	None.
6.......	150	450	None. Only care is to guard against too much intake air.
9.......	160	640	Not to allow ashes and clinker to work up along walls.
32......	250	500	None.
19......	300	300	None.
16......	400	400	Taking out too many ashes, allowing bed to drop too low.

PLANTS BURNING BITUMINOUS COAL.

3.......	200	400	Little trouble. If bed hangs it may burn man in falling.
37......	200	400	Occasional difficulty in breaking down the bridge; then must drive the bar through with the sledge.
39......	370	1,100	Impossibility of properly breaking up and settling clinker so as to maintain even fire.
5.......	1,000	4,000	None.
12......	2,500	2,500	None.

PLANTS BURNING LIGNITE.

15......	100	100	Clinker.
11......	250	500	None unless fire is allowed to drop.
38......	300	300	None. Same lining in producer for 5 years. Cleaning out hard mixture of tar and ash deposit in gas collector and scrubber inlet pipe is dirty work and takes a little time.

PLANTS BURNING WOOD.

13......	150	150	Filling of gas main by tar. This is burned out occasionally. To be done away with by improved gas cleaner.
26......	200, 280	1,040	Tar.

DOWN-DRAFT PLANTS.

PLANTS BURNING BITUMINOUS COAL.

29......	200	200	Poor gas and sometimes trouble with clinkers.
27......	250	500	Producer can not be cleaned while running.
35......	250	1,000	Heat and dirt.
28......	375, 500	1,750	Hard clinker.
22......	500	500	Chopping down clinker.
8.......	800	3,200	None.
23......	1,500	6,000	None.
24......	1,500	9,000	Hard clinker. Loss of coke and thin fire to start next run.

DOUBLE-ZONE PLANT.

PLANT BURNING BITUMINOUS COAL.

8.......	500	500	None.

OIL-GAS PLANT.

PLANT BURNING CRUDE OIL.

30......	....	....	The rapidity with which generator must be cleaned. Reserve gas supply limited.

TIME BETWEEN PERIODS OF DRAWING PRODUCER FIRES.

The following tabulation gives data regarding time between periods of drawing producer fires in the various types of gas producers:

Data regarding time between periods of drawing producer fires in various types of gas producers.

Plant No.	Horsepower of each gas generator.	Total horsepower of plant.	Remarks of owner or operator regarding time between periods of drawing producer fires.
UP-DRAFT PLANTS.			
PLANTS BURNING ANTHRACITE COAL.			
14	75	150	20 days.
20	200	....	45 days.
32	250	500	Present fire, 8 months; previous fire, 4 years.
19	300	300	Once a year.
36	300	300	Indefinite.
1	350	700	14 days. One producer each week.
16	400	400	Not drawn.
PLANTS BURNING BITUMINOUS COAL.			
3	200	400	365 days.
37	200	400	10 days.
33	250	....	2 days.
39	370	1,100	Fires not drawn except for repairs.
21	650, 1,000	3,650	12 months.
5	1,000	4,000	Every third day.
12	2,500	2,500	6 months to 1 year.
PLANT BURNING LIGNITE.			
4	100	100	120 days.
38	300	300	About once a year.
PLANT BURNING WOOD.			
26	200, 280	1,040	3 months or more.
DOWN-DRAFT PLANTS.			
PLANTS BURNING BITUMINOUS COAL.			
29	200	200	3 months.
20	250	....	45 days.
27	250	500	6 days; necessary to cut unit out when coke is 80 to 90 per cent burned out and producer badly clinkered.
35	250	1,000	12 days.
28	375, 500	1,750	14 days
22	500	500	Weekly.
8	800	3,200	10 to 15 days.
23	1,500	6,000	13 to 17 days.
24	1,500	9,000	15 to 21 days.
DOUBLE-ZONE PLANTS.			
PLANTS BURNING BITUMINOUS COAL.			
2	250	250	Do not draw fires.
8	500	500	10 to 15 days.
PLANT BURNING LIGNITE.			
31	200	....	2 years.
OIL-GAS PLANT.			
PLANT BURNING CRUDE OIL.			
30	....	....	Every 8 hours.

METHODS OF SCRUBBING AND CLEANING GAS.

Data supplied by owners and operators regarding methods of scrubbing and cleaning the gas in the various types of gas producers follow.

Data regarding methods of scrubbing and cleaning gas in various types of gas producers.

UP-DRAFT PLANTS.

PLANTS BURNING ANTHRACITE.

Plant No.	Horse-power of each gas generator.	Replies from owners or operators.
6	150	Counter flow wet coke tower scrubber and excelsior dry scrubber.
20	200	Coke.
34	250	Water spray dripped through coke.
19	300	Static washer.
36	300	Static scrubber. Centrifugal tar extractor.
1	350	Wood cross sections through which water flows.
16	400	Coke between 1 and 2 inches with water sprays.

PLANTS BURNING BITUMINOUS COAL.

3	200	Latticed spray scrubbers, rotary tar extractors and purifiers.
37	200	Rotary water-jet scrubber.
17	250	Smith type "F" tar extractors connected in series.
33	250	Preliminary wet scrubber, counter current. Rotary scrubber and dry scrubber.
39	370	Cooled in first pass of wet scrubber, passed through wooden checker work which is sprayed with water. Passed through centrifugal tar extractor to holder.
25	300, 400	Cooling, spraying, and tar extracting. Wood centrifugal tar extractor on one plant and Smith static tar extractor on the other.
21	650, 1,000	Ssaler washers. Four coolers with water spray. No filling in coolers.
5	1,000	Buffalo scrubber. No dry scrubber used. Gas for heating purposes and not for engines.
12	200 300 2,500	Smith type "F" spun-glass tar extractors.

PLANTS BURNING LIGNITE.

4	100	Cooling with water and passing through mechanical scrubber.
11	250	Gas passes through baffles of 2-inch by 4-inch timbers placed on edge, set close together, 14 feet high. Four streams of water flush each scrubber. Gas then passes through tar extractor.
38	300	Spray scrubber and mechanical gas washer (tar extractor).

PLANTS BURNING WOOD.

26	200, 280	Static scrubber, centrifugal tar extractor.

DOWN-DRAFT PLANTS.

PLANTS BURNING BITUMINOUS COAL.

29	200	Cooled with water spray. Passed through coke with water running over it and down through 6 or 8 feet of straw.
27	250	Scrubbers, 28 inches in diameter by 14 feet, each with seven water sprays. Each exhauster has 16 sprays, throwing water at 40 pounds' pressure against first impeller running at 3,500 revolutions per minute.
35	250	Passed through wet and dry scrubber, coke and excelsior.
28	375, 500	Passed through wet scrubber and then exhauster and dry scrubber. One wet scrubber filled with excelsior, other with coke. Both dry scrubbers filled with excelsior.
22	500	Cooling in boiler, wet scrubber and dry scrubber.
8	800	Static wet scrubbers with water sprays, dry scrubber with excelsior.
23	1,500	Gas runs down center of cooler through spray, up inside of shell, through sprays and through a rotary scrubber.
24	1,500	Plant A—through vertical boiler, spray cooler, and centrifugal scrubber. Plant B—gas is not clean except from one producer. Special use. Cooled centrifugal tar scrubber and dry cleaner.

Data regarding methods of scrubbing and cleaning gas in various types of gas producers—Continued.

DOUBLE-ZONE PLANTS.

PLANTS BURNING BITUMINOUS COAL.

Plant No.	Horse-power of each gas generator.	Replies from owners or operators.
2	250	Wet scrubber.
8	500	Static wet scrubber with water sprays. Dry scrubber with excelsior.

PLANT BURNING LIGNITE.

Plant No.	Horse-power of each gas generator.	Replies from owners or operators.
31	200	Water spray.

OIL-GAS PLANTS.

PLANTS BURNING CRUDE OIL

Plant No.	Horse-power of each gas generator.	Replies from owners or operators.
10	400	Static scrubber filled with baffles cleaning gas to about 1½ grains per cubic foot. Buffalo Forge centrifugal scrubber cleaning to about 0.02 grain per cubic foot. Used 2 years without sign of need of change.
30	....	Gas is forced through three turns, 10 inches by 18 feet, against a gravity flow of water running over baffle plates.

TIME BETWEEN RENEWALS OF SCRUBBER MATERIAL.

The owners and operators questioned furnished little information relating to the time between changes in the scrubber material used in the producer-gas installations. The few plants that reported indicated the periods to be as follows:

Time between renewals of scrubber material in various types of producer-gas plants.

UP-DRAFT PLANTS.

Plants burning anthracite coal.

Plant No.	Time between renewals of scrubber material, months.
20	12
16	12
6	[a] 24 to 36 [b] 3 to 6
9	6
34	48

Plants burning bituminous coal.

Plant No.	Time between renewals of scrubber material, months.
33	8
12	2 to 5
21	3

Plant burning lignite.

Plant No.	Time between renewals of scrubber material, months.
11	Never.

Plant burning wood.

Plant No.	Time between renewals of scrubber material, months.
26	Never.

[a] Coke.

[b] Excelsior.

DOWN-DRAFT PLANTS.

Plants burning bituminous coal.

Plant No.	Time between renewals of scrubber material, months.
29	6 to 12
35	[a] 12 [b] 0.5
22	[c] 2 [d] 0.5
8	1¼
24	1 to 1½

[a] Coke. [b] Excelsior. [c] Top. [d] Bottom.

OPINIONS REGARDING SCRUBBING AND CLEANING RESULTS.

The various owners and operators were asked whether the scrubbing and cleaning results in their producers were satisfactory, and if not, why. The data obtained on this point are presented below:

Data regarding satisfactoriness of scrubbing and cleaning results in various types of gas producers.

UP-DRAFT PLANTS.

PLANTS BURNING ANTHRACITE.

Plant No.	Horsepower of each gas generator.	Remarks of owner or operator regarding satisfactoriness of scrubbing and cleaning results.
6	150	Satisfactory.
9	160	Do.
20	200	Do.
32	250	Do.
34	250	Do.
19	300	Do.
36	300	Do.
1	350	Fairly.
16	400	Satisfactory.

PLANTS BURNING BITUMINOUS COAL.

Plant No.	Horsepower of each gas generator.	Remarks of owner or operator regarding satisfactoriness of scrubbing and cleaning results.
3	200	Satisfactory.
37	200	Very satisfactory.
33	250	Not satisfactory. Considerable tar remains in gas and clogs engine valves.
39	370	As satisfactory as ever observed in pressure up-draft producer using bituminous coal.
25	300, 400	Satisfactory.
21	650, 1,000	Does very well, though gas carries tar after cleaning.
5	1,000	For heating purposes; not for gas engines.
12	200 300 2,500	Entirely satisfactory.

PLANTS BURNING LIGNITE.

Plant No.	Horsepower of each gas generator.	Remarks of owner or operator regarding satisfactoriness of scrubbing and cleaning results.
4	100	Satisfactory.
7	100	Do.
11	250	Satisfactory. Tar gives no trouble whatever in engine.
38	300	Satisfactory. Except occasional cleaning each 3 to 4 months. Tar is of soft paraffin nature, but contains a resinal substance, and when dry is hard to remove.

PLANTS BURNING WOOD.

Plant No.	Horsepower of each gas generator.	Remarks of owner or operator regarding satisfactoriness of scrubbing and cleaning results.
13	150	Not satisfactory. Are preparing more complete arrangements to recover acids and tars for utilization and for elimination of condensed matter in gas mains.
26	200, 280	Not satisfactory. On account of moisture and tar.

Data regarding satisfactoriness of scrubbing and cleaning results in various types of gas producers—Continued.

DOWN-DRAFT PLANTS.

PLANTS BURNING BITUMINOUS COAL.

Plant No.	Horsepower of each gas generator.	Remarks of owner or operator regarding satisfactoriness of scrubbing and cleaning results.
29.......	200	Reasonably satisfactory. Slight trouble from tar.
27.......	250	Does not remove lampblack.
35.......	250	Not satisfactory. Considerable dirt and dust get by.
28.......	375,500	Pipes have to be cleaned out occasionally.
22.......	500	Not entirely satisfactory. Soot and lampblack not completely removed.
8.......	800	Satisfactory.
23.......	1,500	Entirely satisfactory.
24.......	1,500	Plant A—satisfactory; plant B—new outfit; good so far (5 months).

DOUBLE-ZONE PLANTS.

PLANTS BURNING BITUMINOUS COAL.

2.......	250	Not satisfactory. Does not remove tar.
8.......	500	Satisfactory.

PLANT BURNING LIGNITE.

31.......	200	Satisfactory.

OIL-GAS PLANTS.

PLANTS BURNING CRUDE OIL.

10.......	400	No tar.
30.......	....	Not satisfactory. Very difficult to get out the floating carbon.

MANNER IN WHICH TAR GIVES TROUBLE.

Data obtained from the operators and owners in response to a query as to the way in which tar gave trouble are presented in the tabulation following:

Data regarding tar troubles in various types of gas producers.

UP-DRAFT PLANTS.

PLANTS BURNING BITUMINOUS COAL.

Plant No.	Horsepower of each gas generator.	Statement of owner or operator regarding tar troubles.
3.......	200	No operating trouble.
37.......	200	Clogs the governor valve on engine.
33.......	250	Choking and sticking of engine valve.
39.......	370	Clogs mains, fittings, engine regulators, and inlet valve passages. Frequent cleaning of all gas piping required.
25.......	300,400	No trouble.
21.......	650,1,000	Most in gas house in handling. Have little trouble where gas is used.
5.......	1,000	Chokes pipes between cooling tower and washers.
12.......	200 300 2,500	No trouble at all.

Data regarding tar troubles in various types of gas producers—Continued.

UP-DRAFT PLANTS—Continued.

PLANTS BURNING LIGNITE.

Plant No.	Horse-power of each gas generator	Statement of owner or operator regarding tar troubles.
4.......	100	No serious trouble.
11.......	250	No trouble at all.
38.......	300	No trouble with any part of engine except governor valve chamber and mixing valve chamber. Must be cleaned every 3 months.

PLANTS BURNING WOOD.

13.......	150	Clogging mains.
26.......	200, 280	In long gas lines to engine and admission valves.

DOWN-DRAFT PLANTS.

PLANTS BURNING BITUMINOUS COAL.

29.......	200	Makes engine valve stick and causes some trouble in exhauster.
27.......	250	Produced only when starting new fire. Clogs ports in exhauster.
35.......	250	Clogs scrubbers.
28.......	375, 500	No trouble.
8.......	800	Do.
23.......	1,500	Do.
24.......	1,500	Labor for cleaning water seal, stopping sprays in cooler and scrubber. Small amount collects at burners in few months, interfering with the seating of mushroom valves on the line.

DOUBLE-ZONE PLANTS.

PLANTS BURNING BITUMINOUS COAL.

2.......	250	Sticking intake valves.
8.......	500	No trouble.

PLANT BURNING LIGNITE.

31.......	200	On valves of engines.

OIL-GAS PLANT.

PLANT BURNING CRUDE OIL.

30.......	....	Causes throttle, poppet, and inlet valves and stack to choke.

TROUBLE CAUSED BY LAMPBLACK.

Data supplied by owners and operators regarding trouble caused by lampblack follow:

Data regarding trouble from lampblack in various types of producers.

UP-DRAFT PLANTS.

PLANTS BURNING BITUMINOUS COAL.

Plant No.	Horsepower of each gas generator.	Remarks of owner or operator regarding trouble caused by lampblack.
3.......	200	Stops water outlet in spray scrubbers.
37......	200	By collecting in and reducing area of gas mains.
33......	250	Lampblack mixing with tar.
39......	370	No trouble observed. If any, it is mixed with tar.
25......	300, 400	No trouble.
21......	650, 1,000	Choking flues in producers and coolers.
12......	200, 300, 2,500	No trouble.

DOWN-DRAFT PLANTS.

PLANTS BURNING BITUMINOUS COAL.

27......	250	Mixes with oil and gums engine valves.
35......	250	Clogs scrubbers.
23......	375, 500	Deposits in pipes.
22......	500	In engine cylinder. Deposits on inlet valves.
8.......	800	No trouble.
24......	1,500	Do.

DOUBLE-ZONE PLANT.

PLANT BURNING BITUMINOUS COAL.

8.......	800	No trouble.

OIL-GAS PLANTS.

PLANTS BURNING CRUDE OIL.

10......	400	Gradually chokes gas line and soots up gas valves. Requires cleaning every 6 months; 10-hour job.
30......	....	Clogs gas main. Works by piston rings and mixes with lubricating oil.

RELATION OF TAR OR LAMPBLACK IN ENGINE TO COST OF LUBRICATION.

In response to a query as to whether the cost of engine lubrication is materially increased by the presence of tar or lampblack in the engine, the following information was supplied:

Data regarding relation of lampblack in gas-producer engine to cost of lubrication.

UP-DRAFT PLANTS.

PLANT BURNING ANTHRACITE COAL.

Plant No.	Horsepower of each gas generator.	Remarks of owner or operator in response to the query: "Is the cost of engine lubrication increased by the presence of tar or lampblack?"
9	160	No.

PLANTS BURNING BITUMINOUS COAL.

3	200	No.
33	250	Yes.
39	370	Not determined.
25	300, 400	No.
21	650, 1,000	Not greatly.
5	1,000	Yes.

PLANT BURNING LIGNITE.

4	100	No.

PLANT BURNING WOOD.

26	200, 280	Yes.

DOWN-DRAFT PLANTS.

PLANTS BURNING BITUMINOUS COAL.

29	200	No.
22	500	Not materially.
8	800	No.
23	1,500	Yes.
24	1,500	Think not.

PLANT BURNING LIGNITE.

38	300	No. No lampblack ever seen.

DOUBLE-ZONE PLANTS.

PLANTS BURNING BITUMINOUS COAL.

2	250	No.
8	500	Do.

PLANT BURNING LIGNITE.

31	200	Yes.

OIL-GAS PLANTS.

PLANTS BURNING CRUDE OIL.

10	400	No.
30	....	No. Use cheap oil.

BURNER TROUBLE CAUSED BY TAR AND LAMPBLACK.

Data supplied by owners and operators regarding trouble from tar or lampblack clogging the gas burners in the various types of gas producers follow; the number at the beginning of each paragraph indicates the plant that supplied the information:

5. None.
9. None.
12. None.
13. Burner is piece of 8-inch iron pipe; clogged sometimes; cleaned once per month.
17. Latest burner gives no trouble.
22. None.
24. No trouble. Remove some lump tar from burners about once each two months.
25. Gas so cleaned that no trouble is experienced.
30. None.
32. None.
35. Burners clog.
38. None.

TROUBLE FROM SULPHUR IN PRODUCER GAS.

Data regarding trouble from sulphur in producer gas as experienced by the various owners and operators follow:

Data regarding trouble from sulphur in gas of various types of gas producers.

UP-DRAFT PLANTS.

PLANTS BURNING ANTHRACITE COAL.

Plant No.	Horsepower of each gas generator.	Replies from owners or operators.
9	160	None.
20	200	Do.
18	300	Trouble; by clogging passages where velocity of flow is changed and by eating out sheet-steel parts.
19	300	None.
PLANTS BURNING BITUMINOUS COAL.		
3	200	Cuts water-cooled valves.
37	200	None.
17	250	When producer has stood over for 20 hours sulphur is noticed by men at furnaces.
25	300, 400	None.
21	650, 1,000	Trouble.
5	1,000	Trouble; causes distress among employees.
12	2,500	Trouble; high sulphur gives unpleasant fumes in factory.
PLANTS BURNING LIGNITE.		
11	250	Exhaust valve seats become pitted.
38	300	None, except that engine exhaust has deteriorated roof metal work, guy ropes, etc.
DOWN-DRAFT PLANTS.		
PLANTS BURNING BITUMINOUS COAL.		
29	200	None.
20	250	Do.
35	250	Do.
28	375, 500	Do.
22	500	Trouble; acts on valves and pipes, and particularly on exhaust.
8	800	No serious trouble.
23	1,500	None.
24	1,500	Do.

PROPORTION OF SULPHUR ALLOWABLE IN FUEL.

The owners and operators were asked as to what percentage of sulphur could be present in the gas-producer fuel without causing inconvenience to the workmen. Summarized data supplied in the replies follow:

Data regarding percentage of sulphur allowable in fuel for producer-gas plants.

UP-DRAFT PLANTS.

PLANT BURNING ANTHRACITE COAL.

Plant No.	Horsepower of each gas generator.	Remarks of owner or operator regarding percentage of sulphur allowable in gas-producer fuel.
19......	300	1.5 to 1.75 per cent.

PLANTS BURNING BITUMINOUS COAL.

17......	250	3 per cent.
33......	250	2 per cent.
21......	650, 1,000	Not over 1 per cent.
12......	2,500	1.5 per cent.

PLANT BURNING LIGNITE.

38......	300	Do not know per cent, but have seen sulphur plainly visible on fuel and had no trouble.

DOUBLE-ZONE PLANTS.

PLANTS BURNING BITUMINOUS COAL.

2.......	250	1 per cent has never caused trouble; none higher used.
8.......	500	Approximately 1.5 per cent.

AUXILIARY POWER USED.

Many producer plants require a small amount of auxiliary power for the operation of tar extractors, blowers, pumps, exhausters, air compressors, etc. This total auxiliary power varies with the different conditions of plant operation and is, as a rule, less for plants operating with anthracite coal than for plants using tar-producing fuels. An idea of the amount of auxiliary power required may be had from the data presented in the table following, which shows the amount installed and the amount actually used.

Auxiliary power used at various producer-gas plants.

UP-DRAFT PLANTS.

Plant No.	Fuel used.	Total horsepower of plant.	Total horsepower of installed auxiliaries.	Proportion of total plant horsepower— Installed in auxiliaries.	Proportion of total plant horsepower— Actually used by auxiliaries.
				Per cent.	*Per cent.*
38	Lignite	300	6.0	2.0	1.3
3	Bituminous coal	400	4.0	1.0	1.0
37	do	400	18.0	4.5	3.3
16	Anthracite coal	400	15.0	3.75	....
11	Lignite	500	25.0	5.0	5.0
33	Bituminous coal	500	30.0	6.0	6.0
32	Anthracite coal	500	10.0	2.0	....
18	do	600	15.0	2.5	1.3
12	Bituminous coal	1,000	17.0	1.7	1.7
26	Wood	1,040	70.0	6.7	1.9
39	Bituminous coal	1,100	165.0	15.0	6.2
25	do	1,400	113.0	8.0	3.9
34	Anthracite coal	1,500	10.0	0.7	0.7
12	Bituminous coal	2,500	65.0	2.6	2.6
5	do	4,000	105.0	2.6	2.0
Average				4.3	2.8
DOWN-DRAFT PLANTS.					
29	Bituminous coal	200	....	....	3.0
20	do	250	5.0	2.0	....
22	do	500	10.0	2.0	2.0
27	do	500	48.0	9.5	5.0
35	do	1,000	100.0	10.0	10.0
8	do	3,200	170.0	4.0	2.9
23	do	6,000	160.0	2.7	2.0
24	do	9,000	450.0	5.0	1.7
Average				5.0	3.8

So much depends on the details of the installation, the number of hours of service per week, and the commercial demands regarding reliability that there may be little relation between the actual auxiliary requirements of different plants. A summary of the data presented in the preceding table follows:

Summary of data regarding auxiliary power used at various producer-gas plants.

Kind of plant.	Proportion of total plant power— Installed in auxiliaries. Maximum.	Installed in auxiliaries. Minimum.	Installed in auxiliaries. Average.	Actually used by auxiliaries. Maximum.	Actually used by auxiliaries. Minimum.	Actually used by auxiliaries. Average.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
Up draft	15.0	0.7	4.3	6.2	0.7	2.8
Down draft	10.0	2.0	5.0	10.0	2.0	3.8

TROUBLE FROM PRODUCER AUXILIARIES.

Data regarding serious trouble with producer auxiliaries were requested from the various owners and operators. A summary of the information furnished follows:

Data regarding trouble from auxiliaries used with gas producers.

UP-DRAFT PLANTS.

PLANTS BURNING ANTHRACITE COAL.

Plant No.	Horsepower of each gas generator.	Remarks of owner or operator regarding serious trouble with producer auxiliaries.
14	75	No trouble.
6	150	Do.
9	160	Do.
20	200	Do.
32	250	Do.
18	300	When fan-type exhausters were used on outlet of producer they were eaten away by sulphurous compounds.
19	300	No trouble.
36	300	Do.
16	400	Do.

PLANTS BURNING BITUMINOUS COAL.

3	200	No trouble.
37	200	Do.
33	250	Trouble; scrubber gets choked with tar.
39	370	Tar extractors become clogged and passages of scrubbers using excessive power cause high pressures at top of producers, causing gas to leak around poke hole, causing distress to operators. Further, under such conditions flues in fire develop rapidly when pressure is relieved at time poke holes are opened for inspection.
25	300, 400	No trouble.
21	650, 1,000	Do.
5	1,000	Do.
12	2,500	No trouble except that due to mechanical defect in steam engine driving the gas pump.

PLANTS BURNING LIGNITE.

4	100	No trouble.
15	100	Do.
11	250	Do.
38	300	Do.

PLANTS BURNING WOOD.

13	150	No trouble.
26	250	Do.

DOWN-DRAFT PLANTS.

PLANTS BURNING BITUMINOUS COAL.

29	200	No trouble.
20	250	Do.
27	250	Trouble on starting new fires a little coal tar is produced, clogging exhauster. Cleaned every 3 weeks.
35	250	No trouble.
28	375, 500	Do.
22	500	Do.
8	800	Do.
23	1,500	Do.
24	1,500	Do.

Data regarding trouble from auxiliaries used with gas producers—Continued.

DOUBLE-ZONE PLANTS.

PLANTS BURNING BITUMINOUS COAL.

Plant No.	Horsepower of each gas generator.	Remarks of owner or operator regarding serious trouble with producer auxiliaries.
2	250	No trouble.
8	500	Do.

PLANT BURNING LIGNITE.

31	200	No trouble.

OIL-GAS PLANTS.

PLANTS BURNING CRUDE OIL.

10		No trouble.
30		No trouble; wash water pump collects tar on runner shaft.

TIME REQUIRED TO BRING GAS PRODUCERS TO FULL GAS PRODUCTION AFTER STAND-BY.

A summary of the information furnished by owners and operators regarding the time required to bring the various types of producers to condition of normal demand after a stand-by follows:

Data regarding time required to bring various types of gas producers to condition of normal demand after a stand-by.

UP-DRAFT PLANTS.

PLANTS BURNING ANTHRACITE COAL.

Plant No.	Horsepower of each gas generator.	Remarks of owner or operator regarding time required for producer to reach full production after stand-by.
6	150	1 hour after 18 hours.
32	250	½ hour after 14 hours.
34	250	3 hours Monday forenoon.
18	300	½ hour after 13 hours.
19	300	1 hour after 14 hours.
36	300	40 minutes after 14 hours.
1	350	½ hour.

PLANTS BURNING BITUMINOUS COAL.

3	200	½ hour after 12 hours.
37	200	1 hour after 14 hours.
17	250	1 hour to come in on main line after 10 hours.
33	250	1 hour after 12 hours.
39	370	1 to 2 hours after 12 hours.
25	300,400	1 hour after 12 hours.
21	650,1,000	¾ hour after 13 hours.
5	1,000	1½ hours after 24 hours.
12	2,500	½ hour to 1 hour after 14 hours.

Data regarding time required to bring various types of gas producers to condition of normal demand after a stand-by—Continued.

UP-DRAFT PLANTS—Continued.

PLANTS BURNING LIGNITE.

Plant No.	Horsepower of each gas generator.	Remarks of owner or operator regarding time required for producer to reach full production after stand-by.
4.......	100	20 minutes after 12 hours.
7.......	100	1 hour after 12 hours.
11......	250	2 hours after 12 hours.
38......	300	20 minutes to ½ hour after 8 hours; 1 hour to 1½ hours after 1 week.

PLANT BURNING WOOD.

26......	200, 280	½ hour after 24 hours to 1 week.

DOWN-DRAFT PLANTS.

PLANTS BURNING BITUMINOUS COAL.

29......	200	Service continuous.
27......	250	Can start engine in 15 minutes after 12 hours, but takes about 1 hour to get to working well.
23......	1,500	4 hours after 32 hours.
24......	1,500	30 minutes to 1 hour after 12 hours.

DOUBLE-ZONE PLANTS.

PLANTS BURNING BITUMINOUS COAL.

2.......	250	½ hour after 18 hours.
8.......	500	1 hour after 16 hours.

OIL-GAS PLANT.

PLANT BURNING CRUDE OIL.

30......	....	Producer shows good efficiency after 10 minutes' operation.

STAND-BY FUEL.

Many controversies have arisen regarding the stand-by losses in producer-gas plants. What this percentage may be under test and what it is under commercial operating conditions are two widely different values. Several writers on the subject are in the habit of allowing per stand-by producer hour from 3 to 6 per cent of the fuel charged in the producer per operating hour.

An attempt was made to procure figures covering commercial operation in several plants but the returns were so greatly at variance that no deductions of value could be presented. The figures reported show stand-by percentages ranging from 3 to 33.

QUANTITY OF WATER USED BY PRODUCER PLANTS.

The quantity of water actually required in the operation of a producer-gas plant and the quantity used by the plants reporting seem to bear little relation to each other. This variance is largely due to the fact that most of the plants reporting seem to have ample water supply in close proximity so that the water cost is small.

Obviously, the quantity of water required in both the generation of the gas and in the cleaning process will vary according to the type of producer plant, the character of the fuel used, the method of operation, and the efficiency of the scrubbing devices.

QUANTITY OF VAPORIZER WATER USED.

The quantity of vapor used by the gas generators per pound of fuel fired was not reported by the operators queried. Previous investigations have indicated that for up-draft plants this figure usually runs from 0.7 to 1 pound of water for the vaporizer to each pound of anthracite coal fired. With bituminous coals, lignites, peats, wood, and other fuels the quantity of water required by the vaporizer will vary greatly with the percentage of moisture contained in the fuel. With bituminous coals in up-draft plants this figure seems to be not far from that required for anthracite coals. The average for 20 bituminous coals taken at random shows 0.7 pounds of water per pound of coal.

On the other hand, a series of investigations with one grade of fuel in an up-draft plant showed the quality of the gas to vary little for a range of vaporizer consumptions from 0.7 pound to 1.12 pounds.

In using peat it has been found that if the peat contains 25 or 30 per cent moisture no steam is necessary in the operation of the plant.

In one plant in this country operating on lignite the supply of available water is seriously limited. The moisture contained in the lignite is, however, sufficient to more than make up the vaporization requirements so that in the process of cooling the gases enough water vapor is condensed to more than offset the losses. This plant is, therefore, actually increasing its water supply during operation.

During a series of tests with a down-draft producer at the Pittsburgh station of the Bureau of Mines, the average consumption of water by the vaporizer was as follows:

Consumption of water by vaporizer in down-draft producer of Bureau of Mines.

Fuel.	Water per pound of fuel as fired, pounds.
Bituminous coal	0.23
Lignite	.00
Peat	.00

QUANTITY OF SCRUBBER WATER USED.

The quantity of water required for scrubbing the gas in the operation of a producer-gas plant is reported by different writers as follows:

(*a*) 1 gallon per 40 cubic feet of gas washed.

(*b*) 3.3 gallons per brake horsepower-hour.

(*c*) 10 to 15 pounds per pound of fuel used in the gas generator.

If these quantities are reduced to the basis of cubic feet of water required per 1,000 cubic feet of gas washed the range is as follows:

(*a*) 3.3 cubic feet per 1,000 cubic feet of gas.

(*b*) 6 cubic feet per 1,000 cubic feet of gas.

(*c*) 2.6 to 3.9 cubic feet per 1,000 cubic feet of gas.

The latter figures indicate an average of approximately 4 cubic feet of water required for scrubbing 1,000 cubic feet of gas.

During the operation of the testing station of the Bureau of Mines the quantities of scrubber water averaged approximately as follows:

9.7 cubic feet per 1,000 cubic feet of gas with the up-draft plant, including the centrifugal tar extractor.

10.5 cubic feet per 1,000 cubic feet of gas with the down-draft plant.

Figures supplied by the operators of several plants are as follows:

Quantities of scrubber water used in different gas producers.

Plant No.	Fuel.	Operation.	Quantity of scrubber water per pound of fuel.
			Pounds.
37	Bituminous coal	Up-draft	76
17	do	do	26
39	do	do	67
25	do	do	36
25	do	do	31
22	do	Down-draft	116
8	do	Down-draft and double-zone.	95

If these figures are reduced to cubic feet of water per 1,000 cubic feet of gas washed by assuming that 1 pound of bituminous coal is equivalent to 61, 90, and 83 cubic feet of gas for up-draft, down-draft, and double-zone producers, the values are as follows:

Quantities of scrubber water used per 1,000 cubic feet of gas scrubbed.

Plant No.	Water per 1,000 cubic feet of gas scrubbed, cubic feet.
37	19.9
17	6.8
39	17.5
25	9.5
25	8.1
22	20.6
8	16.9
Average	14.2

The range between the temperature of the water entering and that of the water leaving the scrubber seems to depend more on the quantity of water available than on any predetermined temperature as the proper one for the water leaving the scrubber.

The prevailing temperature for the water entering the scrubber is 50° to 90° F., and for the water leaving it 75° to 200° F., the average being about 110° F.

The temperature rise recorded for the different plants is as follows:

Rise in temperature of scrubber water at different plants.

Plant No.	Temperature rise, °F.
14	45
3	8
37	10
15	50
1	155
26	37
39	44
25	45
21	70
22	15
8	55
2	10
10	40

QUANTITY OF WATER REQUIRED TO COOL ENGINE.

The quantity of water required in engine cooling is reported by various writers as follows:

(*a*) For single-acting engines, 5 to 7 gallons per horsepower-hour.

(*b*) For large double-acting engines, 4.5 to 5.5 gallons per horsepower-hour for cylinders, stuffing boxes, valves, etc., and 1.75 to 2.25 gallons per horsepower-hour for pistons and pistons rods; or a total ranging from 6.25 to 7.75 gallons per horsepower-hour.

(*c*) Twenty-five to fifty pounds per brake horsepower-hour, or 3 to 6 gallons.

(*d*) For a producer-gas engine, single-acting and of less than 200 horsepower, the normal consumption of water per brake horsepower-hour at full load is 5.5 gallons for cooling the engine.

(*e*) For engines of 2 to 1,000 horsepower, for cylinder covers and stuffing boxes, 5.5 to 6.5 gallons; pistons and rods, 2 to 2.5 gallons; boxes, seats, and exhaust valves, 1 to 1.75 gallons. Total, 9 to 11 gallons per brake horsepower-hour.

(*f*) With a temperature range of 90° F., 45 pounds or 5.5 gallons will be required per horsepower-hour. In large engines the consumption is smaller, or about 4.25 gallons. To be on the safe side the cooling water may be estimated at 5.5 to 8 gallons.

If the various values be reduced to the common basis of cubic feet of water per horsepower-hour, the figures are as follows:

Quantity of engine-cooling water required in various plants on basis of cubic feet per horsepower-hour.

Report.	Cooling water per horsepower-hour, cubic feet.
a	0.67 to 0.93
b	.83 to 1.03
c	.40 to .80
d	.73 to .73
e	1.20 to 1.47
f	.67 to 1.07
Average	.75 to 1.00

The Bureau of Mines figures show an average of 0.82 cubic foot per horsepower-hour for a three-cylinder, single-acting engine of 250 horsepower.

The wide variation in practice is shown by the following figures from plant operators:

Quantity of engine-cooling water required at different producer-gas plants.

Plant No.	Cooling water per horsepower-hour, cubic feet.
37	3. 36
25	2. 80
35	2. 56
22	1. 01
8	2. 18
8	2. 56

The inlet temperatures reported for the cooling water range from 50° to 90° F. and the outlet temperatures from 86° to 160° F., the average being about 115° F.

LUBRICANTS USED.

The quantity of oil required per horsepower-hour varies with the character of the installation and the method of operation. For full load, 24-hour service, the proportion per horsepower-hour is of course greater than for a plant running under light load for a 9-hour or 10-hour day. Some of the figures given by the engine manufacturers for the quantity of engine oil required are as follows:

1. For a 200-horsepower engine, the oil amounts to 1.25 gallons per 10-hour day, or 0.625 gallon per 1,000 horsepower-hours.
2. For a 65-horsepower engine, the oil used is $\frac{1}{2000}$ gallon per brake horsepower-hour, or 0.500 gallon per 1,000 horsepower-hours.
3. For a 100-horsepower engine, inclosed crank case, 0.5 gallon per 10 hours, or 0.500 gallon per 1,000 horsepower-hours.

4. For a 140-horsepower engine, inclosed crank case, 1 gallon per 10 hours, or 0.715 gallon per 1,000 horsepower-hours.

5. For a 125-horsepower engine, inclosed crank case, 1 quart per 10 hours, or 0.200 gallon per 1,000 horsepower-hours.

The average of these quotations is 0.508 gallon per 1,000 horsepower-hours. The plant operators reported their commercial requirements to be as follows:

Quantity of oil used in lubricating engines in various gas producers.

Plant No.	Horsepower of engines.	Length of service per day.	Cylinder oil used per 1,000 horse-power-hours.	Engine oil used per 1,000 horse-power-hours.	Other lubricants used per 1,000 horse-power-hours.
		Hours.	*Gallons.*	*Gallons.*	*Gallons.*
4	100	8	2.0		
7	100				1.25
14			1.8	1.3	
3	40, 160	16	2.8		
16	190	24		1.0	
11	500	24	1.25		
9	80, 160, 200, 375	12	1.5	3.0	
12	200, 500	24		1.26	
39	300	24	.75	.4	.8
25	750	10	.5	.17	.07
21	125		.13	.4	.1
35	150, 250, 300, 600		.5	1.0	
22	500	10	.5	.6	.14
23	500, 1,000	10	.4	.6	
24	300, 2,000	24	2.7	5.3	.7
2	115, 300, 750	24	.25	.5	
30		24	1.25		
Average			1.17	1.11	.51

The average of a number of returns from the operators of reciprocating steam engines indicates the consumption of cylinder oil and engine oil to be approximately the same and to equal 0.13 gallon each per horsepower-hour. On this basis the oil consumption of gas engines seems to be approximately eight or nine times as much as that of reciprocating steam engines. This difference is perhaps not surprising, as the lubricating requirements of the gas engine are much more severe than those of the steam engine, but the ratio seems rather high.

PREIGNITIONS AND THEIR CAUSES.

Data supplied by owners and operators regarding preignitions and their causes follow. The numbers preceding each paragraph refer to plant numbers assigned by the author.

Are preignitions frequent and troublesome.—To what do you attribute them?

UP-DRAFT PLANTS.

Plants burning anthracite coal.

1. Yes. Carbon deposits.
6. No.
9. No. Most trouble from too high compression.

14. No. Excessive hydrogen.
16. No.
18. No.
19. No.
20. No. Excessive hydrogen.
32. No.
34. No. Hydrogen.
36. No.

Plants burning bituminous coal.

3. No, if good coal is used and valves are tight. Hydrogen, bad gas, and leaky valves.
12. No.
21. None.
25. No. Overhanging fire or hot particles of carbon from lubrication.
33. No.
37. No.
39. No.

Plants burning lignite.

4. Yes; at times. Carbon deposits, etc.
7. No.
11. Only for first hour after starting. Excessive hydrogen.
15. No.
38. Very seldom. To a hot spot or "chimney" in producer forming high hydrogen or to small particles of tar being sucked off governor valve and holding fire in cylinder. Stopped by throttling gas valve and then sweeping cylinder with air.

Plants burning wood.

26. Sticking valves from tar.

DOWN-DRAFT PLANTS.

Plants burning bituminous coal.

8. No.
20. No. Excessive hydrogen.
22. Yes. Dirt, incandescent points, too rich mixture.
23. No.
24. No.
27. Had trouble for a while, but seldom now . Holes or channels in fires.
28. After water gas run and when air inlet valve leaks. Too rich gas.
29. No. Do not have any.
35. No. Carbon.

DOUBLE-ZONE PLANTS.

Plants burning bituminous coal.

2. No.
8. No.

Plants burning lignite.

31. At times. Sticking valves.

OIL-GAS PLANTS.

Plants burning crude oil.

10. No. Very little. Small portions of lampblack carrying a spark.
30. No. Excess hydrogen under high compression.

CHANGING OR CLEANING IGNITERS.

The time interval for reliable use of engine igniters is of course more or less dependent on the constituents of the gas, the degree of cleanliness, the amount of sulphur, the engine construction, and the care in operation. The table following indicates the reported practice in this connection:

Data regarding changing or cleaning of igniters in gas producers.

UP-DRAFT PLANTS.

PLANTS BURNING ANTHRACITE COAL.

Plant No.	Period of operation each day.	Period before igniter is cleaned or changed.
	Hours.	
14	24	Cleaned each month; lasts 4 years.
19	24	2 months.
16	24	¾ month.
6	24	¼ month.
9	24	⅓ month.
1	5-24	1½ months.
34	24	½ month.
18	11½	Do.

PLANTS BURNING LIGNITE OR BITUMINOUS COAL.

4	8	¾ month.
7	11	2 months.
3	24	¼ month.
37	10	⅓ month.
33	14	Do.
12	24	¼ to ⅓ month.
39	24	2 months.
25	10	8 months.
21	10	2 months.

DOWN-DRAFT PLANTS.

PLANTS BURNING BITUMINOUS COAL.

29	24	Years.
27	10	¼ month.
35	10	⅓ month.
22	10	4 months.
8	24	2 months.
23	24	3 months.

DOUBLE-ZONE PLANTS.

PLANTS BURNING BITUMINOUS COAL OR LIGNITE.

31	24	1 month.
2	24	Do.

GAS PRESSURES AND DISTANCES FOR GAS DELIVERY.

It is interesting to note that in practically all of these plants the gas is delivered at a short distance from the producer. The reports from 36 plants show a range of distances of 15 to more than 3,000 feet. Twenty-four plants deliver their gas at distances not exceeding 100 feet and only 5 deliver gas to a distance exceeding 500 feet.

None of these plants carries gas at any considerable pressure. The range seems to be from a slightly less than atmospheric to 2½ pounds higher than atmospheric. In the majority of instances the positive pressure ranges from 1 to 8 ounces.

RECOVERY OF BY-PRODUCTS FROM WASTE LIQUORS FROM SCRUBBERS, WASHERS, ETC.

To the query regarding recovery of by-products from waste liquors from scrubbers, washers, etc., the answers were all "None recovered" with the exception of four. Two operators report a special effort to procure the tar; one stated that he recovered the by-products, but gave no further information, and the fourth reported "Not yet."

COMMERCIAL USE MADE OF TAR PRODUCED.

The various owners and operators were asked what commercial use was made of the tar produced in their plants and what price was received for it. The data supplied are presented below. The numbers preceding each paragraph refer to plant numbers assigned by the author.

2. None.
3. None.
4. None.
5. None.
7. None.
9. No tar.
11. No market for tar from lignite.
12. Fired under boilers in main boiler plant. Saves 5 tons of coal per day, equal to $10.
13. Distilled for wood oil, creosote, and pitch.
14. None recovered.
17. Run back into producer.
21. Burned under boiler to make steam for use in gas plant.
23. No tar.
24. Mixed with coal and burned in hand-fired boiler. Not satisfactory.
25. Utilized in steam-boiler plant. The tar from one of the producers returns to the producer and is gasified.
26. Returned to producer by spreading on fuel.
28. None.
30. None.
35. None.
37. Burned under boiler.

38. One barrel per day goes to Puget Sound with scrubber water. Formerly burned with fuel oil under boilers, but caused some trouble on account of water. Will soon sell it or burn it again. Worth about $1.10 per barrel if burned under boiler.

39. Originally provision was made for introducing the tar into the producer combustion zone, but the tar was too stiff to handle.

COMMERCIAL UTILIZATION OF CARBON RESIDUE FROM OIL-GAS PRODUCERS.

Only two replies were received to the query, Is the carbon residue from oil-gas producers commercially utilized, and if so, how? Plant 10 reported that the residue was used under boilers, and plant 30 reported that it was used as fuel in blast furnaces.

ENRICHMENT OF PRODUCER GAS.

The various owners and operators were asked whether they knew of any plants that enriched the producer gas. Replies to this inquiry indicated that the enriched producer gas was used to some degree, but not generally. Three or four companies were reported to be using this enriched gas for illuminating purposes. One plant enriched the producer gas with gasoline vapor for use in brazing.

TEMPERATURE REQUIRED AT FURNACE.

Data received from the various owners and operators regarding the temperature required at the furnace follow.

10. About 1,400° F.
12. 950 to 1,050° F. in leers.
13. 650° F.
17. 1,700° F.
21. 1,300 to 2,200° F.
24. As high as 1,500° F.
25. 1,500 to 2,000° F.

SPARE CAPACITY AND POWER ACTUALLY AVAILABLE.

Practically the same general rules that govern the installation of spare boiler units in steam plants control the installation of spare producer units. The principal points that have to be considered are reliability of plant and continuity of service.

The need of reserve boiler units is, however, much greater under normal operating conditions on account of the necessity of frequent boiler cleaning. Although the intermittent type of producer must be cleaned at regular intervals, depending in length on the percentage of ash in the fuel, the continuous type may be run for years without the fires being drawn if clinker troubles are not excessive.

An examination of the data at hand shows no relation between the daily hours of service and the installation of spare units. Of the plants from which reports were received, ranging in capacity from

100 to 9,000 horsepower, about one-third have reserve units of 20 to 50 per cent of the total installed power.

Ten years ago most gas producers for power purposes were overrated in capacity.

This overrating was the natural result of the blind use of European figures and became for a time a serious matter. Although the rating of such plants is far more conservative to-day, still an examination of the operating reports of 27 plants reveals the fact that approximately one-third of them have considerably less than 100 per cent of their capacity available. One instance is reported in which only 60 per cent of the rated capacity could be realized. The average percentage of available capacity for the plants that are below rating is 75.

On the other hand, 6 of the 27 plants show a capacity considerably above normal rating. The maximum is 155 per cent, and the average for the 6 is 132 per cent.

USES OF PRODUCER-GAS PLANTS.

To indicate the varied applications of producer gas and producer-gas power a brief tabulation is presented giving the information received from the 39 companies that reported. The numbers preceding the paragraphs refer to plant numbers.

1. Lights and power (5 to 24 hours per day).
2. Commercial lighting and power (24 hours per day).
3. Manufacture of acid phosphate (24 hours per day).
4. City water company (8 hours per day).
5. General factory purposes; also drying molds and covers.
6. Light and power (24 hours per day).
7. Milling flour (11 hours per day).
8. Induction motor drive (24 hours per day).
9. Lighting and power (24 hours per day).
10. Street and house lighting, ice making, refrigeration, etc. (16 hours per day).
11. Grinding cottonseed cake into meal for export.
12. Motor drives (24 hours per day, 6½ days per week). Gas also used for leers in glass bottling factory.
13. For heating wood-distillation retorts (22 to 24 hours per day).
14. For operating blowing engines and for oprating pyrites furnace (24 hours per day.
15. Light and power (14 hours per day).
16. Mining and concentrating iron ore (24 hours per day).
17. Hardening and annealing.
18. Machine shop, electric power (11½ hours per day).
19. Factory lighting and power, motor-driven ammonia compressor for raw-water ice making (summer, 24 hours per day; winter, 8 to 16 hours).
20. Electric lighting and power (24 hours per day).
21. Driving machinery by rope transmission from engine to line shaft (10 hours per day); also forge, annealing, and hardening.
22. Manufacturing (10 hours per day).

3. Manufacture of plate glass, power (24 hours per day, 5½ days per week).

24. Excitation of direct-current electric generator, lights and general plant auxiliary; alternating-current motors driving grinding and polishing machines, 1,000 to 1,200 kilowatts on each alternating-current machine. (Continuous, 24 hours per day, 156 hours per week.) Also melting glass in furnaces and glass annealing.

25. Power in manufacturing plant (10 hours per day). Also hardening and annealing.

26. Power by direct drive and also direct-connected to direct-current generators (7 months, 24 hours per day; 5 months part load, 24 hours per day). Exhaust gas used to produce steam at 60 pounds pressure on vaporizers connected to engine exhausts.

27. Factory power.

28. Electric power for phosphate mining. Principal load is motor-driven centrifugal pumps (156 hours per week).

29. Oil mill, electric power (24 hours per day).

30. Pumping (24 hours per day).

31. Electric power (24 hours per day) and concentration of acid.

32. Machine-shop power (10 hours per day) and drying linings in foundry ladles.

33. Electric shop drive (14 hours).

34. Power in paper mill to drive beaters and pumps; also belted generators (24 hours per day, 6 days per week).

35. Power for manufacturing purposes, shops (10 hours per day); also for heating carbonizing furnaces.

36. Power for manufacturing (10 hours per day).

37. Shop power and lighting (9 hours per day).

38. Driving ammonia compressor and electric generator for power and light (4½ months, 24 hours per day with not to exceed one stop per 20 to 30 days for cleaning and adjustments in the summer months; 7 to 8 months, 10 to 20 hours per day). Producer has had fires drawn not to exceed five times in five years.

39. Operating motor-driven centrifugal pumps, rotating and shaking screens and elevators, and for lighting purposes. Also conveyors, rotating dry kilns, washers, shops, etc. (24 hours per day).

RESPONSE OF PRODUCER TO SUDDEN CHANGES IN DEMAND.

Following are tabulated data relative to the response of various types of gas producers to sudden changes in demand:

Data regarding response of various types of gas producers to sudden changes in demand.

UP-DRAFT PLANTS.

PLANTS BURNING ANTHRACITE COAL.

Plant No.	Horsepower of each gas generator.	Reply of owner or operator to the query, "Does the gas producer respond readily to sudden changes in demand?"
1	350	Yes.
6	150	Yes; for engine load fluctuation. Starting a second engine takes about 20 minutes before gas is strong.
9	160	Reasonably so.
14	75	Load is nearly constant. Believe it would be slow.
16	400	Yes.
18	300	Do.
19	300	Do.
20	200	Do.
32	250	Producer is so large that it instantly covers requirements.
34	250	One alone does not. Good on full number.
36	300	Yes.

Data regarding response of various types of gas producers to sudden changes in demand—Continued.

UP-DRAFT PLANTS—Continued.

PLANTS BURNING BITUMINOUS COAL.

Plant No.	Horsepower of each gas generator.	Reply of owner or operator to the query, "Does the gas producer respond readily to sudden changes in demand?"
3	200	Yes; if fuel bed is in good shape.
5	1,000	Yes.
12	2,500	Do.
17	250	Do.
21	650, 1,000	No trouble.
25	300, 400	Yes.
33	250	No.
37	200	Yes; as long as fires are kept regular.
39	370	No.

PLANTS BURNING LIGNITE.

4	100	Yes; when working well.
7	100	Yes.
11	250	Do.
15	100	Do.
38	300	Do.

PLANTS BURNING WOOD.

13	150	Yes; about 5 minutes required for increase in volume.
26	200, 280	Yes.

DOWN-DRAFT PLANTS.

PLANTS BURNING BITUMINOUS COAL.

6	800	Perfectly.
20	250	Yes.
22	500	Have none.
23	1,500	Yes.
24	1,500	Do.
27	250	No.
28	375, 500	Proper care required.
29	200	No.
35	250	Demand is steady.

DOUBLE-ZONE PLANTS.

PLANTS BURNING BITUMINOUS COAL.

2	250	Yes.
8	500	Perfectly.

PLANT BURNING LIGNITE.

31	200	Yes.

PERIOD OF CONTINUOUS OPERATION.

The data following were furnished in answer to a query regarding the period of operation of gas producers.

Period of continuous operation of gas producers.

UP-DRAFT PLANTS.

Plant No.	Horse-power of each gas generator.	Length of service each day.	Time in continuous operation.
		Hours.	*Days.*
PLANTS BURNING ANTHRACITE COAL.			
14	75	24	20
6	150	24	Continuous.
32	250	3 to 7	Do.
34	250	24	Do.
19	300	24	50
36	300	10	Continuous.
1	350	5 to 24	7
16	400	24	330
PLANTS BURNING BITUMINOUS COAL.			
17	250		6
33	250	14	2
25	300 400	10	Continuous.
39	370	24	3
21	650 1,000	10	Continuous.
5	1,000	16	6
12	2,500	24	180 to 360
PLANTS BURNING LIGNITE.			
4	100	8	200 to 300
7	100	11	6
15	100	14	3
11	250	24	Continuous.
38	300	24	Do.
PLANTS BURNING WOOD.			
13	150		7
26	200 280	24	7

DOWN-DRAFT PLANTS.

Plant No.	Horse-power of each gas generator.	Length of service each day.	Time in continuous operation.
PLANTS BURNING BITUMINOUS COAL.			
29	200	24	Continuous.
20	250	24	30
27	250	10	6
35	250	10	6
28	375 500	24	14
22	500	10	6
8	800	24	10 to 15
23	1,500	24	15
24	1,500	24	15 to 20

DOUBLE-ZONE PLANTS.

Plant No.	Horse-power of each gas generator.	Length of service each day.	Time in continuous operation.
PLANT BURNING BITUMINOUS COAL.			
2	250	24	Continuous.
PLANT BURNING LIGNITE.			
31	200	24	730

RELIABILITY AND LENGTH OF SERVICE OF PLANTS.

The following queries were included in the general request for information sent to owners and operators of gas producers: Has the plant proved reliable? If not, what is the cause of failure? How many years has the plant been in service? The data supplied follow.

Data regarding reliability and length of service of producer-gas plants.

UP-DRAFT PLANTS.

PLANTS BURNING ANTHRACITE COAL.

Plant No.	Horsepower of each gas generator.	Total horsepower of plant.	Reply of owner or operator to queries.
14	75	150	Yes. In service 7 years.
19	300	300	Yes.
6	150	450	In service 3 years.
32	250	500	Yes. In service 8 years.
9	160	640	Reasonably so. Have had a few minor troubles, but nothing serious. In service 7 years.
1	350	700	No. Too many little things that may happen to cause a shutdown, such as change of quality of gas and poor ignition. In service 3½ years.
20	200	....	Yes, after some minor changes.
18	300	....	Yes. In service 4 years.

PLANTS BURNING BITUMINOUS COAL.

3	200	400	Yes, very reliable. Plant competes with a hydroelectric plant that offers current at less than 2 cents per kilowatt-hour. A part of the plant is run with motors, but a majority of it is run from a main line shaft. The manufacturing processes require steady power 24 hours a day, 7 days a week. In service 9 years.
17	250	500	Yes.
33	250	....	No. Tar and gas stick valves of engine and choke scrubber and pipes.
39	370	1,100	No. Irregularity in thermal quality of gas. Choking of gas mains and engine regulators with tar. Engines are undoubtedly rated too high. Improved mixing valves and gas-inlet passages would probably result in increased capacity. However, considerable expense would be involved. Plant has been superseded by 1,000-kilowatt steam turbine installed in 1911. In service 2 years.
25	300,400	1,400	Yes. One unit in service 3 years, and one unit 2 years.
12	200,300,2,500	3,500	Yes. Power units in service 3 years; fuel unit, 1 year.
21	650,1,000	3,650	After first year. At first had some trouble on account of tar and with the washing equipment. Since October, 1913, have been using natural gas. In service 4 years.
5	1,000	4,000	Producers were operated 3 years continuously and were discontinued, but are still in good condition.

PLANTS BURNING LIGNITE.

4	100	100	Producer was bought for 100 horsepower, but at present does not have capacity of 90.
7	100	....	Yes. In service 5 years.
15	100	100	No. Unit has not been run during the past 12 months. Failure thought to be due to prejudice on the part of engineer and helpers, or possibly the attention required was too tedious compared with the steam plant.
38	300	300	Yes. Most trouble and expense has been with exhaust piping. This trouble was overcome by installing two short sections of water-jacketed exhaust pipes connecting into fire-brick lined steel shell placed between the twin engines and containing a stack of cast-iron water-heating spiders, or star-shaped sections, which heat all jacket water from 130° to 200° F. About one-half of this hot water is used for industrial purposes and the balance for humidifying the producer ash-pit air. In service 5 years and 4 months.
11	250	500	Yes. In service 5 years.

Data regarding reliability and length of service of producer-gas plants—Continued.

UP-DRAFT PLANTS—Continued.

PLANTS BURNING WOOD.

Plant No.	Horsepower of each gas generator.	Total horsepower of plant.	Reply of owner or operator to queries.
13......	150	150	Yes. After finding out how to use wet wood. When plant was first started used up a large quantity of good dry wood on hand. Plant then put on very green wet wood, requiring much reconstruction of method of operation. In service intermittently for 2 years.
26......	200, 280	1,040	Yes.

DOWN-DRAFT PLANTS.

PLANTS BURNING BITUMINOUS COAL.

29......	200	200	"All my figures have been destroyed, as we gave up the engine 2 years ago and have since had a fire. We are using hydroelectric power altogether and find it much more successful."
20......	250	....	Yes, after some minor changes.
22......	500	500	Yes. In service 7½ years.
35......	250	1,000	Yes. Company is now changing over to hydroelectric power. Producers and engines have been in service over 10 years and for many reasons are not using power economically, owing largely to rapid plant growth and necessarily poor power-plant location.
28......	375, 500	1,750	One pair of producers in service 6 years and one pair 2 years.
8......	800	3,200	Yes. In service 6 years.
23......	1,500	6,000	Yes. In service 4 years.
24......	1,500	9,000	To certain extent. Lack of knowledge of requirements to make good quality gas, including proper grade and kind of coals, grates, steam and air supply, and method of firing and cost of apparatus.

DOUBLE-ZONE PLANT.

PLANT BURNING BITUMINOUS COAL.

2......	250	250	Fairly. Most trouble from poor grade of coal. In service 2½ years.

PUBLICATIONS ON FUEL TECHNOLOGY.

A limited supply of the following publications of the Bureau of Mines is temporarily available for free distribution. Requests for all publications can not be granted, and applicants should limit their selection to publications that may be of especial interest to them. Requests for publications should be addressed to the Director, Bureau of Mines, Washington, D. C.

BULLETIN 2. North Dakota lignite as a fuel for power-plant boilers, by D. T. Randall and Henry Kreisinger. 1910. 42 pp., 1 pl., 7 figs.

BULLETIN 4. Features of producer-gas power-plant development in Europe, by R. H. Fernald. 1910. 27 pp., 4 pls., 7 figs.

BULLETIN 5. Washing and coking tests of coal at Denver, Colo., July 1, 1908, to June 30, 1909, by A. W. Belden, G. R. Delamater, J. J. Groves, and K. M. Way. 1910. 62 pp., 1 fig.

BULLETIN 6. Coals available for the manufacture of illuminating gas, by A. H. White and Perry Barker, compiled and revised by H. M. Wilson. 1911. 77 pp., 4 pls., 12 figs.

BULLETIN 16. The uses of peat for fuel and other purposes, by C. A. Davis. 1911. 214 pp., 1 pl., 1 fig.

BULLETIN 24. Binders for coal briquets, by J. E. Mills. 56 pp., 1 fig. Reprint of United States Geological Survey Bulletin 343.

BULLETIN 27. Tests of coal and briquets as fuel for house-heating boilers, by D. T. Randall. 44 pp., 3 pls., 2 figs. Reprint of United States Geological Survey Bulletin 366.

BULLETIN 28. Experimental work conducted in the chemical laboratory of the United States fuel-testing plant at St. Louis, Mo., January 1, 1905, to July 31, 1906, by N. W. Lord. 51 pp. Reprint of United States Geological Survey Bulletin 323.

BULLETIN 31. Incidental problems in gas-producer tests, by R. H. Fernald, C. D. Smith, J. K. Clement, and H. A. Grine. 29 pp., 8 figs. Reprint of United States Geological Survey Bulletin 393.

BULLETIN 33. Comparative tests of run-of-mine and briquetted coal on the torpedo boat *Biddle*, by W. T. Ray and Henry Kreisinger. 50 pp., 10 figs. Reprint of United States Geological Survey Bulletin 403.

BULLETIN 36. Alaskan coal problems, by W. L. Fisher. 1911. 32 pp., 1 pl.

BULLETIN 39. The smoke problem at boiler plants, a preliminary report, by D. T. Randall. 31 pp. Reprint of United States Geological Survey Bulletin 334, revised by S. B. Flagg.

BULLETIN 40. The smokeless combustion of coal in boiler furnaces, with a chapter on central heating plants, by D. T. Randall and H. W. Weeks. 188 pp., 40 figs. Reprint of United States Geological Survey Bulletin 373, revised by Henry Kreisinger.

BULLETIN 49. City smoke ordinances and smoke abatement, by S. B. Flagg. 1912. 55 pp.

BULLETIN 55. The commercial trend of the producer-gas power plant in the United States, by R. H. Fernald. 1913. 93 pp., 1 pl., 4 figs.

BULLETIN 56. First series of gold-dust tests in the experimental mine, by G. S. Rice, L. M. Jones, J. K. Clement, and W. L. Egy. 1913. 115 pp., 12 pls., 28 figs.

Bulletin 58. Fuel-briquetting investigations, July, 1904, to July, 1912, by C. L. Wright. 1913. 277 pp., 21 pls., 3 figs.

Bulletin 76. United States coals available for export trade, by Van. H. Manning. 1914. 15 pp., 1 pl.

Bulletin 85. Analyses of mine and car samples of coal collected in the fiscal years 1911 to 1913, by A. C. Fieldner, H. I. Smith, A. H. Fay, and Samuel Sanford. 1914. 444 pp., 2 figs.

Bulletin 116. Methods of sampling delivered coal and specifications for the purchase of coal for the government, by G. S. Pope. 1916. 64 pp., 5 pls., 2 figs.

Technical Paper 2. The escape of gas from coal, by H. C. Porter and F. K. Ovitz. 1911. 14 pp., 1 fig.

Technical Paper 5. The constituents of coal soluble in phenol, by J. C. W. Frazer and E. J. Hoffman. 1912. 20 pp., 1 pl.

Technical Paper 8. Methods of analyzing coal and coke, by F. M. Stanton and A. C. Fieldner. 1913. 42 pp., 12 figs.

Technical Paper 9. The status of the gas producer and of the internal-combustion engine in the utilization of fuels, by R. H. Fernald. 1912. 42 pp., 6 figs.

Technical Paper 10. Liquefied products of natural gas; their properties and uses, by I. C. Allen and G. A. Burrell. 1912. 23 pp.

Technical Paper 16. Deterioration and spontaneous combustion of coal in storage, a preliminary report, by H. C. Porter and F. K. Ovitz. 1912. 14 pp.

Technical Paper 34. Experiments with furnaces for a hand-fired return tubular boiler, by S. B. Flagg, G. C. Cook, and F. E. Woodman. 1914. 32 pp., 1 pl., 4 figs.

Technical Paper 35. Weathering of the Pittsburgh coal bed at the experimental mine near Bruceton, Pa., by H. C. Porter and A. C. Fieldner. 1914. 35 pp., 14 figs.

Technical Paper 37. Heavy oil as fuel for internal-combustion engines, by I. C. Allen. 1913. 36 pp.

Technical Paper 38. Wastes in the production and utilization of natural gas, and methods for their prevention, by Ralph Arnold and F. G. Clapp. 1913. 29 pp.

Technical Paper 45. Waste of oil and gas in the Mid-Continent fields, by R. S. Blatchley. 1914. 54 pp., 2 pls., 15 figs.

Technical Paper 49. The flash point of oils—methods and apparatus for its determination, by I. C. Allen and A. S. Crossfield. 1913. 31 pp., 2 figs.

Technical Paper 50. Metallurgical coke, by A. W. Belden. 1913. 48 pp., 1 pl., 23 figs.

Technical Paper 55. The production and use of brown coal in the vicinity of Cologne, Germany, by C. A. Davis. 1913. 15 pp.

Technical Paper 57. A preliminary report on the utilization of petroleum and natural gas in Wyoming, by W. R. Calvert, with a discussion of the suitability of natural gas for making gasoline, by G. A. Burrell. 1913. 23 pp.

Technical Paper 63. Factors governing the combustion of coal in boiler furnaces; a preliminary report, by J. K. Clement, J. C. W. Frazer, and C. E. Augustine. 1914. 46 pp., 26 figs.

Technical Paper 64. The determination of nitrogen in coal, a comparison of various modifications of the Kjeldahl method with the Dumas method, by A. C. Fieldner and C. A. Taylor. 1915. 25 pp., 5 figs.

Technical Paper 65. A study of the oxidation of coal, by H. C. Porter. 1914. 30 pp., 12 figs.

Technical Paper 74. Physical and chemical properties of the petroleums of California, by I. C. Allen, W. A. Jacobs, A. S. Crossfield, and R. R. Mathews. 1914. 38 pp., 1 fig.

Technical Paper 76. Notes on the sampling and analysis of coal, by A. C. Fieldner. 1914. 59 pp., 6 figs.

TECHNICAL PAPER 89. Coal-tar products and the possibility of increasing their manufacture in the United States, by H. C. Porter, with a chapter on coal-tar products used in explosives, by C. G. Storm. 1915. 21 pp.

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Bulletin 110

DEPARTMENT OF THE INTERIOR
FRANKLIN K. LANE, SECRETARY
BUREAU OF MINES
VAN. H. MANNING, DIRECTOR

CONCENTRATION EXPERIMENTS WITH THE SILICEOUS RED HEMATITE OF THE BIRMINGHAM DISTRICT, ALABAMA

BY

JOSEPH T. SINGEWALD, JR.

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CONCENTRATION EXPERIMENTS WITH THE SILICEOUS RED HEMATITE OF THE BIRMINGHAM DISTRICT, ALA.

By JOSEPH T. SINGEWALD, Jr.

INTRODUCTION.

The possible value of the red hematite iron ore of the southern Appalachian States, if some practicable method of concentrating it could be devised, has long been recognized. The ore is very low grade, and although in enormous quantities, particularly in the States of Alabama, Tennessee, and Georgia, is only at a few points rich enough to work, and even the best of it ranks among the lowest grade iron ores being mined in the United States. A cheap method of beneficiating this ore would make enormous quantities available and vastly increase the iron-ore resources of the country. However, this low-grade ore in effect constitutes a potential iron reserve, and as long as ample quantities of ore can be obtained elsewhere the question of its utilization does not demand immediate notice.

Where these ores are in part of workable grade, however, a problem of more immediate importance arises which, from considerations of economy and prevention of waste in their mining, ought to be solved or, at least, attacked as quickly as possible. Where these ores are being worked they are usually intimately associated with lower-grade material that in mining is left behind. The difficulty and expense of going into ground broken by old workings makes the recovery of this lower-grade ore practically impossible and it is lost forever. Success in concentrating the red ores, therefore, would save material that is now being irretrievably lost. The successful large-scale concentration of low-grade ore in the last few years on the Lake Superior iron ranges should encourage the attack of similar problems in the Birmingham district.

While studying iron-mining methods in the United States for the Bureau of Mines, Dwight E. Woodbridge, consulting mining engineer of the bureau, was impressed with the importance of this problem, and believed it merited the attention of the bureau. Accordingly, Mr. Woodbridge had Mr. W. J. Penhallegon, general superintendent of ore mines and quarries of the Republic Iron & Steel Co., of Birmingham, send the author of this report five samples of the unworkable siliceous ores of the district. The results of concentration experiments with these ores made a more thorough and systematic investigation desirable. In consequence, the author subsequently collected samples in the district, and these became the basis of the experimental work described in this report. The results are published by the Bureau of Mines as a part of its efforts to increase efficiency in the utilization of mineral resources.

The experiments were conducted in the geological laboratory of the Johns Hopkins University, Baltimore, Md., with the aid of Raymond Leibensperger, mining assistant. The chemical analyses were made by A. C. Fieldner, chemist, of the Bureau of Mines.

The author wishes to express his appreciation of the courtesies extended to him by the mining companies of the district, and particularly to Mr. W. J. Penhallegon, of the Republic Iron & Steel Co. Thanks are also due Prof. W. B. Clark, director of the geological laboratory of the Johns Hopkins University, for the unrestricted use of the facilities of the laboratory.

GENERAL FIELD RELATIONS AND QUALITY OF THE ORES.

The general field relations of the ores of the Birmingham district have been fully described by Burchard and Butts,[a] and somewhat the same ground, with greater emphasis on the industrial conditions of the district, is covered by Phillips.[b] For detailed particulars on these points the reader is referred to these two reports, as no attempt will be made here to duplicate the information they present. For the benefit of those not especially interested in these points, and to make the purpose of this report more intelligible to such to whom the above publications are not available, a summary of the general field relations of the district is given.

EXTENT OF THE DISTRICT.

The Birmingham district is a narrow belt about 75 miles long and 10 miles wide, with a northeast trend. Near the middle of the belt is the city of Birmingham, which stands in a broad anticlinal valley, known as Birmingham Valley, that extends the whole length of the belt. The iron ores lie in the ridges on both sides of the valley, but by far the most important beds are along the east side, in what is known as Red Mountain. Nearly all of the mining in the district has been confined to that part of Red Mountain lying between Morrow Gap on the northeast and Sparks Gap on the southwest, a distance of about 25 miles. This part of the district and the location of the mines from which the samples were collected are shown in Plate I. The area of greatest activity is even smaller, and in the summer of 1914, when the iron industry was at a low ebb, mining was practically confined to that part of Red Mountain between Graces Gap and Sparks Gap, a distance of about 10 miles. All the largest mines of the district are included in this latter area.

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[a] Burchard, E. F., and Butts, Charles, Iron ores, fuels, and fluxes of the Birmingham district, Ala., with chapters on the origin of the ores, by E. C. Eckel: Bull. 400, U. S. Geol. Survey, 1910, 204 pp.

[b] Phillips, W. B., Iron making in Alabama: 3d ed., Geol. Survey of Alabama, 1912, 254 pp.

THE ORE BEDS.

The ores are bedded in the Clinton or Rockwood formation, of Silurian age, which outcrops along Red Mountain, and gives the mountain its name. The beds dip away from the valley at angles ranging from 15° to 45°, but averaging less than 30°. One or more ore beds of greater or less importance are everywhere present in the Rockwood formation, and within that part of Red Mountain of greatest economic importance—that is, between Morrow Gap and Sparks Gap—there are four important beds, known, respectively, in their order from top to bottom, as the Hickory Nut, Ida, Big, and Irondale seams. The Hickory Nut seam is the least important of the four and has received little attention. It comprises 3 to 5 feet of sandy ore or ferruginous sandstone, characterized by abundant fossils of the brachiopod *Pentamerus oblongus*, which, on account of looking like a hickory nut in its hull, has given the bed the name. The bed lies about 12 to 20 feet above the Ida seam and reaches its greatest thickness between Birmingham and Bessemer. The other three seams are described on subsequent pages in connection with the results of the concentration experiments.

MINERALOGICAL COMPOSITION OF THE ORES.

Mineralogically the ores consist of red hematite and include two structural varieties, oölitic and fossil, which usually occur mixed. Usually the nuclei of the oölites are small grains of sand, about which successive layers of iron oxide, and frequently thin layers of silica and aluminous material, have been deposited. The fossil ore consists of fragments of such fossil forms as bryozoans, crinoids, corals, and brachiopods.

In addition to the iron oxide the ores carry varying percentages of calcium carbonate, silica, alumina, magnesium carbonate, and other constituents in minor amounts. Where surface waters have leached out the calcium carbonate the ore is porous and friable, and is known as "soft ore"; in contrast, the unaltered ore is called "hard ore." The "hard ores" naturally form the great bulk of the deposits, and those mined at present range in composition from 32 to 45 per cent iron, 5 to 20 per cent lime, 2 to 25 per cent silica, 2 to 5 per cent alumina, 1 to 3 per cent magnesia, 0.25 to 1.5 per cent phosphorus, from a trace up to 0.5 per cent sulphur, and 0.5 to 3 per cent water.[a] Actually the ores average well under 40 per cent iron, and their value depends chiefly on the relative proportions of lime and silica. Roughly speaking, a self-fluxing ore is one in which the lime is slightly in excess of the silica. As such an ore requires in smelting no addition of limestone, it is equivalent to a higher grade siliceous ore that requires added flux. If the proportion of lime is in excess of that required for a self-fluxing ore, a quantity of siliceous ore can be added. This is done in the district for the most part by using a mixed-charge of limy ore and brown ore.

THE PROBLEM OF CONCENTRATION.

The great bulk of the red ore of the district is high in silica and comparatively low in lime, so that the addition of considerable flux would be necessary in smelting. The iron content of the ore is so low, however, that the iron content of the furnace charge would be reduced enough to make the use of the ores unfeasible. Only those ores, therefore, that are self-fluxing or nearly self-fluxing can be used at present.

How far the ore can depart from the self-fluxing type and still be workable depends entirely on the status of the iron industry. For example, the upper bench of the Big seam at the Spaulding mine is workable when the demand for iron is good and unworkable when the demand slumps. The average composition of this ore is about 35 to 36 per cent iron, 17 to 18 per cent silica, and 12 per cent lime, and it may be regarded as representing the dividing line between ore that is workable and ore that is not workable under average conditions.

Most of the ore of this composition, or better, lies between Birmingham and Bessemer, and is Big seam ore. But, as is explained more fully in the section dealing with the Big seam ores, only the upper part of the Big seam comes within these limits, consequently the lower part is left in the mines. As the old workings cave, the collapsed roof will make unprofitable the future mining of the rest of the bed. It is with reference to the lower bench of the Big seam in this part of the Birmingham district that this investigation is of most importance. For each ton of ore removed in mining at least one ton of the siliceous lower bench is left in the ground, and as the annual production is three to four million tons, the annual loss is at least that much. The total of the ore reserves in the lower bench of the Big seam within the area under consideration is estimated at 146,000,000 tons.[a]

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[a] Burchard, E. F., and Butts, Charles, Iron ores, fuels, and fluxes of the Birmingham district, Alabama, with chapters on the origin of ores, by E. C. Eckels: U. S. Geol. Survey Bull. 400, 1910, p. 27.

An early solution of the problem of beneficiating these lower bench ores is, therefore, to be sought in order to stop the waste of resources that at some future time will undoubtedly have value. Also, the solution of this problem would place among the available iron-ore reserves of the country a vast tonnage of low-grade ores in other parts of Alabama, as well as in Tennessee and Georgia, and wherever the Clinton ores occur.

For the purpose of determining the possibility of removing silica and concentrating iron in these ores, 39 samples were taken from various mines lying along Red Mountain between the Ruffner mine on the northeast and the Fossil mines on the southwest; 2 of these samples were from the Ida seam, 3 from the Irondale, 24 represented the lower bench of the Big seam, and the other 10 were from the upper bench; 23 of the 24 lower bench samples were taken along Red Mountain southwest of Graces Gap; that is, in the mines where this ore is being lost.

EARLIER WORK ON THE BENEFICIATION OF THE CLINTON ORES.

The problem of beneficiating the Clinton ores has been attractive enough to stimulate numerous attacks in the past. Efforts have been directed toward applying some known method of treatment or toward devising some new method especially adapted to these ores. The fact that none of the methods is in use shows that the attempts were not commercially successful. Results of many experiments by private individuals on their own initiative or by private companies have never been made known. Recently an experimental mill, said to have a capacity of about 300 tons in 24 hours, was erected at one of the mines and has made trial runs, but the persons interested refuse to give out any data on the results.

The most extensive series of experiments that have been carried out seem to be those of Phillips,[b] and of Wilkens and Nitze,[c] made nearly 20 years ago.

Results are summarized in Phillips' Iron Making in Alabama, chapter IV, concentration of ores. In the first of these experiments the ore was heated to make it magnetic, and then passed over a magnetic separator; but the experimenter soon found that magnetic separation could be effected directly. Numerous experiments were made on a working scale and about 500 analyses.

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[a] Burchard, E. F., and Butts, Charles, Iron ores, fuels, and fluxes of the Birmingham district, Alabama, with chapters on the origin of the ores, by E. C. Eckel: U. S. Geol. Survey Bull. 400, 1910, p. 182.

[b] Phillips, W. B., Notes on the magnetization and concentration of iron ores: Trans. Am. Inst. Min. Eng., vol. 25, 1896, pp. 399–423; Concentration of low-grade iron ores: Eng. and Min. Jour., vol. 62, 1896, pp. 75, 76. 105, 106, 124, 125, 151.

[c] Wilkens, H. A. J., and Nitze, H. B. C., The magnetic separation of nonmagnetic material: Trans. Am. Inst. Min. Eng., vol. 26, 1897, pp. 351–370. Discussion of this paper by W. B. Phillips, pp. 1089–1093.

Attention was directed chiefly to the low-grade soft ores, and the experiments demonstrated that by crushing such ores to pass a 15-mesh screen, it was entirely feasible, with a ratio of concentration of 2 to 1 and an iron recovery of about 85 per cent, to make concentrates carrying over 50 per cent iron from ores that in their crude state were worthless. The average composition of eight samples of the low-grade crude tested was 38 per cent iron and 42.5 per cent silica. The average composition of the concentrates obtained was 52.34 per cent iron and 21.63 per cent silica, with a ratio of concentration of 1.93 and an average iron recovery of 71.4 per cent. This average concentrate is of somewhat higher grade than the average soft ore now mined. Phillips estimates the cost of producing a ton of 50 per cent iron concentrates under favorable conditions at about $1.35; he says that this kind of ore is worth $1.60 to $1.80 per ton, and that the price for 55 per cent concentrates would be $2 per ton.

The results of four tests of samples of hard ore, as given by Phillips, are as follows:

Results of 4 concentration tests of samples of hard ore.

Sample.	Crude ore.			Concentrates.			Ratio of concentration.	Iron recovery.
	Iron.	Lime.	Insoluble.	Iron.	Lime.	Insoluble.		
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*		*Per cent.*
A	37.60	15.00	16.20	48.70	9.76	10.26	1.82	71.2
B	34.50	17.10	18.04	45.40	11.45	12.25	1.56	84.2
C	31.80	10.79	33.10	43.15	8.80	19.66	2.27	59.7
D	32.80	9.90	33.70	44.50	9.00	17.30	1.73	78.7
Average	34.18	13.20	25.26	45.44	9.75	14.87	1.85	73.5

The first two of these samples represent an almost self-fluxing ore similar to those of the upper bench of the Big seam ores, and although beneficiation has yielded a much higher grade product, ores of this composition can be profitably smelted crude when the demand for iron is strong. Such ores are of slightly higher grade than the upper bench ores of the Spaulding mine, which are just about at the lower workable limit. The last two samples represent ores that more nearly approach the composition of the Ruffner ores described in this report and were decidedly low grade. The results Phillips obtained with these samples were not encouraging, and in this respect are like those the author obtained with the Ruffner ores. Although the concentrate was better than the crude ore, it was not of high enough grade to warrant the cost of treatment.

As the samples tested by Phillips represent two groups of ores of entirely different types, the average of the four samples has no particular significance. It is noteworthy that beneficiation did not reduce the lime content as much as the silica content, so that the concentrates are more nearly self-fluxing than is the crude ore. This result is in accordance with those obtained by the author of this report.

Another point on which the two series of experiments—by Phillips and the author—agree is that the iron content of the concentrates obtained from the soft ores is higher than 50 per cent, whereas that obtained from the hard ores runs below 50 per cent and is usually about 45 per cent.

Unfortunately these experiments throw little light on the amenability to treatment of the siliceous hematites having the composition of those in the lower bench of the Big seam. The upper bench self-fluxing ores are now being worked, and the concentration of the lower bench ores is of immediate importance.

Of great interest, because of the method used being essentially that used in this investigation, are some experiments made in a private laboratory at Wilmington, Del., with what is known as the Moxham-du Pont haloid process. The general results of these experiments have been cited by Burchard[a] and are given here in more detail with the consent of A. J. Moxham. The ore tested was from Pleasant Hill, Ala., and was too low grade for profitable mining. It was ground to pass through a 100-mesh screen and subjected to *float* and sink tests in troughs containing haloid solutions of specific gravity high enough to float the tailings. Three tests of this ore in solutions of different specific gravity gave the results shown below.

Results of 3 concentration tests.

No. of test.	Composition of—						Separation.		Ratio of concentration.	Iron recovered.	Specific gravity of solution.
	Crude ore.		Concentrates.		Tailings.		Concentrates.	Tailings.			
	Iron.	Insoluble.	Iron.	Insoluble.	Iron.	Insoluble.					
	P. ct.	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*		*P. ct.*	
A..........	34.32	44.80	53.32	16.90	17.55	72.90	48.8	51.2	2.05	75.7	3.64
B..........	34.32	44.80	42.40	36.28	14.30	78.60	70.8	29.2	1.41	87.4	3.34
C..........	34.32	44.80	41.10	34.50	6.20	90.76	81.2	18.8	1.23	97.2	3.06

From its composition, this ore probably was a low-grade soft ore, and hence the results can not be compared with those of tests of the siliceous hard ores. The grade of the first concentrate is better than that of the best soft ores, so that the experiments show the possibility of making an easily marketable concentrate from an ore as siliceous as that used and of saving three-fourths of the iron in the crude ore. The cost of crushing to 100 mesh would probably be prohibitive in commercial work. Beneficiation of other low-grade iron ores by this method is being studied, and an experimental plant of commercial size has been erected for testing the Oriskony limonites at the Rich Patch mines in Virginia. If the method finally proves commercially practicable, it will be of great importance in the treatment of siliceous red hematites.

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[a] Burchard, E. F., in Contributions to economic geology, Preliminary report on the red iron ores of east Tennessee, northeast Alabama, and northwest Georgia: U. S. Geol. Survey Bull. 540, 1914, pp. 325–326; Geol. Survey of Tenn., Bull. 16, 1913, p. 161.

Ordinary wet methods of concentration are being used for treating low-grade iron ores on the Lake Superior ranges, and have proved so successful that the tonnage treated is continually increasing. However, these Lake Superior ores differ from the siliceous red hematites under consideration, and hence the results obtained can not be applied to the treatment of Clinton ores. Yet the work has shown that the concentration of iron ores by wet methods is feasible on a large scale.

METHOD OF CONDUCTING THE EXPERIMENTS.

About a pound of each sample was broken in a small Blake crusher and then crushed in a mortar until it all passed through a 20-mesh screen. A small quantity of the crushed ore was taken as a crude ore sample and the rest was subjected to a screen analysis. For this analysis the Tyler standard screen scale sieves were used, the sizes ranging from 20 mesh to 200 mesh. In these screens the openings are so arranged that they increase in the ratio of the square root of 2, the sizes in the screens used are given in the following table:

Mesh dimension of screens.

Mesh.	Opening in—	
	Inches.	Millimeters.
20	0.0328	0.833
28	.0232	.589
35	.0164	.417
48	.0116	.295
65	.0082	.208
100	.0058	.147
150	.0041	.104
200	.0029	.074

As all the sample was finer than 20 mesh, there were eight sizes of material. The seven coarsest were subjected to concentration, but what was finer than 200 mesh was regarded as slimes and was not treated.

To separate the heavier particles, high in iron, from the lighter particles, low in iron and high in silica, a heavy solution, Thoulet solution of a specific gravity of 3.0, was used. Each size of material was treated separately. It was mixed with some of the liquid in a separating funnel; the heavy particles settled to the bottom and the light particles floated, the two portions being then drawn off separately. In this way each sample yielded seven sizes of concentrates and of tailings, besides the slime.

METHOD OF PLOTTING RESULTS.

A graphic representation of the results was prepared by plotting the screen analysis as a broken curve, the sizes being used as abscissas and the percentages of the crushed ore as ordinates. The percentages of concentrates and tailings that each size yielded were also shown graphically. A second curve was plotted with the actual quantity of concentrate produced from each mesh size (percentage of concentrate of each size×percentage of total ore) as ordinates. This second curve clearly shows how much each mesh size contributes to the total concentrates. As the sum of these products gives the total percentage of concentrates from the sample, and as the slimes are known, the percentage of tailings is obtained by subtracting their sum (concentrates+slimes) from 100. These three percentages are represented by column A (see figs. 1–47) the lower part representing the concentrates, the middle lined part, the slimes, and the upper black part, the tailings. Column B in the figures is likewise divided into three corresponding parts, which show the percentage of the total iron in each. From these figures the ratio of concentration can be calculated for the concentrates alone, or of the product obtained by mixing the slimes and concentrates and considering it as the final product of the treatment.

From all but six samples the concentrates were mixed to form a sample of total concentrates, the tailings were mixed to form a total tailings sample, and these two products were analyzed for iron and silica. Also the crude ore sample and the slimes were analyzed. From these analyses and the percentages of concentrates, tailings, and slimes, the percentage of iron recovered in the concentrates can be calculated and also that of the concentrates plus slimes. As the slimes were of about the same composition as the concentrates, adding them to the concentrates much increased the apparent recovery.

In order to show how iron and silica are distributed in the different mesh sizes, and in the concentrates and tailings obtained from them, the concentrates and tailings from each mesh size of six samples were analyzed separately; and the composition of the crude ore of that mesh size, as well as the composition of the total concentrates and tailings, were calculated from the analyses. Lime was determined often enough to show the relation of the lime in the crude ore to that in the concentrates. The pages that follow present the detailed results of these experiments.

RESULTS OF CONCENTRATION TESTS.

THE BIG SEAM.

The Big seam, as its name implies, is the important ore bed of the district, and in June, 1914, was the only one being worked. Its general characteristics are described by Burchard,[a] as follows:

> The thickness of the Big seam varies from 16 to 30 feet. It extends as a traceable unit on Red Mountain for practically the whole length of the mining district. Notwithstanding the great thickness there are rarely more than 10 to 12 feet of good ore in a single bench, and at most places only 7 to 10 feet are mined. Probably the maximum thickness of the bed, without reference to the thickness of the workable part, occurs between Red Gap (near Irondale) and Bald Eagle, although for a mile southwest of Red Gap the bed remains nearly as thick. From northeast to southwest the total thickness of the ore-bearing sediments gradually decreases, without, however, altering greatly the thickness of the workable portion. About the middle of the district the bed becomes separated into two benches, either by a well-defined parting along the bedding plane, or by a shale bed, thin at first, but thickening gradually to the southwest. The middle of the Big seam is the workable part in the northeast end of the district, but the upper bench is of most importance throughout the rest of the area. In the southwest portion of the district the lower bench, which farther northeast is composed of ore that may eventually be mined, becomes a series of strata of lean ore and shale and is consequently of no possible value; and finally the upper bench itself becomes shaly and carries only a very low-grade ore.

From this description of the Big seam it is evident that mining is removing 50 per cent or less of the ore, and the rest is being left in the ground, where the caving of the workings will make future recovery costly if not impossible. In the part of the district now most productive—that is, along Red Mountain from about south of Birmingham to Bessemer—the siliceous ores form the lower part of the seam and the less siliceous and more highly calcareous ores the upper part. Burchard says that in the northeast part of this strip the two benches are separated by a mere parting along the bedding and in many places there is no parting, the dividing line being purely arbitrary; whereas toward the southwest a thin shale parting that gradually increases in thickness comes in. In this part of the district, then, the upper part of the Big seam is being worked and the lower part left in the ground. Consequently most attention was paid to the lower bench ores of this area. However, several samples of upper bench ores were also tested for comparison. On the following pages the tests of upper bench ores are considered apart from those of lower bench ores. Several tests were made of ores not included in either of these groups.

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[a] Burchard, E. F., and Butts, Charles, Iron ores, fuels, and fluxes of the Birmingham district, Alabama, with chapters on the origin of the ores, by E. C. Eckel: U. S. Geol. Survey Bull. 400, 1910, pp. 46, 47.

UPPER BENCH, BIG SEAM.

The character of the upper bench ore gradually changes north of a point about opposite Bessemer. Around Bessemer the ore contains a little more lime than is needed to flux the silica and alumina, and to such ore a siliceous ore can be added to make a self-fluxing charge. Northeastward the silica content rises and the lime content declines, until near Birmingham the ore becomes decidedly siliceous.

Phillips[a] says that hard red ore of good grade has the following composition:

Average composition of good hard ore.

	Per cent.
Water	0.50
Metallic iron	37.00
Silica	13.44
Lime	16.20
Alumina	3 18
Phosphorus	.37
Sulphur	.07
Carbonic acid	12.24

The term self-fluxing, so he states, is applied to those varieties of the limy ore in which the lime content equals the sum of the silica and alumina contents. Hence the analysis above represents a good self-fluxing ore. As the alumina content is usually small, a few per cent, a self-fluxing ore may roughly be considered one in which the lime content is a few per cent in excess of the silica content.

The ore from the mines about Readers Gap averages about 34 to 36 per cent Fe, 10 to 13 per cent SiO_2, about 18 per cent CaO, and nearly 4 per cent Al_2O_3; hence its lime content is higher than that required in a self-fluxing ore. Near Spring Gap the lime content is such that the ores are just about self-fluxing, and at Graces Gap the ores have a considerable excess of silica. Thus the ore of the Songo mine, just south of Spring Gap, averages about 34 per cent Fe, 12.5 per cent SiO_2, and 16 per cent CaO; whereas that from the Spaulding mine, at Graces Gap, averages about 36 per cent Fe, 19 per cent SiO_2, 13 per cent CaO, and nearly 3 per cent Al_2O_3. Northeast of Birmingham the ore grows still more siliceous and at the Ruffner mines averages about 32 per cent Fe, 31 per cent SiO_2, 9 per cent CaO, and 3 per cent Al_2O_3. Thus elimination of silica from upper bench Big seam ore is of importance except in the southwestern part of the district.

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[a] Phillips, W. B., Iron making in Alabama, 3d ed.: Geol. Survey of Alabama, 1912, p. 35.

TABULATED RESULTS.

The results of the concentration tests of the 39 samples of ore collected from various mines are shown in the tables following:

Table 1.—*Results of crushing and separating samples from various mines.*

SAMPLE 1. ISHKOODA MINE.

Screen analysis.			Separation.		Concentrates.
Size of mesh.	Quantity taken for separation.		Concentrates.	Tailings.	
	Grams.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
Through 20, on 28	66.3	21.8	64.5	35.5	14.1
28, on 35	74.2	24.5	68.1	31.9	16.7
35, on 48	54.1	17.8	66.0	34.0	11.7
48, on 65	26.2	8.6	57.1	42.9	4.9
65, on 100	24.8	8.2	53.6	46.4	4.4
100, on 150	18.3	6.0	57.7	42.3	3.5
150, on 200	10.5	3.5	60.9	39.1	2.1
200	29.2	[a] 9.6	....	....	0.0
	303.6	100.0	....	....	57.4

Total concentrates	per cent	57.4
Slimes	do	9.6
Tailings	do	33.0
		100.0

SAMPLE 2. SPAULDING MINE.

Size of mesh.	Grams.	Per cent.	Concentrates, per cent.	Tailings, per cent.	Concentrates, per cent.
Through 20, on 28	129.5	28.2	68.9	31.1	19.4
28, on 35	105.8	22.8	73.4	26.6	16.7
35, on 48	76.3	16.5	75.2	24.8	12.4
48, on 65	41.7	9.0	75.0	25.0	6.8
65, on 100	33.8	7.3	73.4	26.6	5.4
100, on 150	26.0	5.6	75.5	24.5	4.2
150, on 200	17.6	3.8	89.9	10.1	3.4
200	31.6	[a] 6.8	....	....	0.0
	462.3	100.0	....	....	68.3

Total concentrates	per cent	68.3
Slimes	do	6.8
Tailings	do	24.9
		100.0

SAMPLE 3. SPAULDING MINE.

Size of mesh.	Grams.	Per cent.	Concentrates, per cent.	Tailings, per cent.	Concentrates, per cent.
Through 20, on 28	119.4	25.6	68.9	31.1	17.6
28, on 35	103.9	22.3	73.1	26.9	16.3
35, on 48	75.7	16.2	75.2	24.8	12.2
48, 65	36.6	7.8	69.6	30.4	5.4
65, on 100	36.7	7.9	70.4	29.6	5.6
100, on 150	32.9	7.0	70.6	29.4	4.9
150, on 200	18.9	4.0	72.2	27.8	2.9
200	43.1	[a] 9.2	....	....	0.0
	467.2	100.0	....	....	64.9

Total concentrates	per cent	64.9
Slimes	do	9.2
Tailings	do	25.9
		100.0

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[a] Slimes.

Table 1.—*Results of crushing and separating samples from various mines*—Continued.

SAMPLE 4. HAMMOND MINE.

Screen analysis.			Separation.		Concentrates.
Size of mesh.	Quantity taken for separation.		Concentrates.	Tailings.	
	Grams.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
Through 20, on 28	107.2	28.3	47.1	52.9	13.3
28, on 35	83.1	22.0	53.9	46.1	11.9
35, on 48	54.2	14.3	60.9	39.1	8.7
48, on 65	28.4	7.5	71.6	28.4	5.4
65, on 100	30.2	8.0	77.4	22.6	6.2
100, on 150	25.8	6.8	78.2	21.8	5.3
150, on 200	15.4	4.1	79.0	21.0	3.2
200	34.2	[a]9.0	....	....	0.0
	378.5	100.0	....	....	54.0

Total concentrates	per cent	54.0
Slimes	do	9.0
Tailings	do	37.0
		100.0

SAMPLE 5. RUFFNER MINE.

Through 20, on 28	52.8	12.5	55.3	44.7	6.9
28, on 35	56.0	13.3	55.7	44.3	7.4
35, on 48	66.4	15.8	50.7	49.3	8.0
48, on 65	63.3	15.0	53.7	46.3	8.1
65, on 100	78.9	18.8	53.4	46.6	10.0
100, on 150	37.1	8.8	67.9	32.1	6.0
150, on 200	19.2	4.5	63.4	36.6	2.9
200	47.8	[a]11.3	....	....	0.0
	421.5	100.0	....	....	49.3

Total concentrates	per cent	49.3
Slimes	do	11.3
Tailings	do	39.4
		100.0

SAMPLE 5. RUFFNER MINE (UNDER 35 MESH).

Through 35, on 48	39.7	19.5	47.7	52.3	9.3
48, on 65	37.0	18.2	52.5	47.5	9.6
65, on 100	47.5	23.4	50.9	49.1	11.7
100, on 150	23.2	11.4	58.3	41.7	6.7
150, on 200	21.2	10.5	62.5	37.5	6.6
200	34.6	[a]17.0	....	....	0.0
	203.2	100.0	....	....	43.9

Total concentrates	per cent	43.9
Slimes	do	17.0
Tailings	do	39.1
		100.0

[a] Slimes.

TABLE 1.—*Results of crushing and separating samples from various mines*—Continued.

SAMPLE 6. RUFFNER MINE.

Screen analysis.			Separation.		Concentrates.
Size of mesh.	Quantity taken for separation.		Concentrates.	Tailings.	
	Grams.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
Through 20, on 28	55.7	13.6	82.3	17.7	11.2
28, on 35	53.2	13.0	72.7	27.3	9.5
35, on 48	61.2	15.0	70.5	29.5	10.6
48, on 65	59.8	14.6	64.1	35.9	9.4
65, on 100	78.9	19.3	56.8	43.2	11.0
100, on 150	36.2	8.8	64.3	35.7	5.7
150, on 200	19.5	4.8	74.6	25.4	3.6
200	44.7	[a] 10.9	....	....	0.0
	409.2	100.0	....	....	61.0

Total concentrates	per cent	61.0
Slimes	do	10.9
Tailings	do	28.1
		100.0

SAMPLE 6. RUFFNER MINE (UNDER 35-MESH).

Through 35, on 48	46.7	18.5	49.3	50.7	9.1
48, on 65	42.8	17.0	48.0	52.0	8.2
65, on 100	65.1	25.8	47.2	52.8	12.2
100, on 150	30.9	11.9	55.5	44.5	6.6
150, on 200	17.1	7.0	69.5	30.5	4.9
200	50.0	[a] 19.8	....	....	0.0
	252.6	100.0	....	....	41.0

Total concentrates	per cent	41.0
Slimes	do	19.8
Tailings	do	39.2
		100.0

SAMPLE 7. FRANK WHITE MINE.

Through 20, on 28	112.2	26.7	65.7	34.3	17.5
28, on 35	91.7	21.8	73.4	26.6	16.0
35, on 48	57.7	13.7	75.7	24.3	10.4
48, on 65	33.3	7.9	86.1	13.9	6.8
65, on 100	35.4	8.4	86.4	13.6	7.3
100, on 150	32.2	7.7	90.2	9.8	6.9
150, on 200	18.5	4.4	88.5	11.5	3.9
200	39.3	[a] 9.4	....	....	0.0
	420.3	100.0	....	....	68.8

Total concentrates	per cent	68.8
Slimes	do	9.4
Tailings	do	21.8
		100.0

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[a] Slimes.

TABLE 1.—*Results of crushing and separating samples from various mines*—Continued.

SAMPLE 8. FRANK WHITE MINE.

Screen analysis.			Separation.		Concentrates.
Size of mesh.	Quantity taken for separation.		Concentrates.	Tailings.	
	Grams.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
Through 20, on 28	110.2	25.7	75.4	24.6	19.4
28, on 35	90.1	21.1	76.9	23.1	16.2
35, on 48	62.2	14.5	79.4	20.6	11.5
48, on 65	34.3	8.0	85.2	14.8	6.8
65, on 100	37.3	8.7	88.1	11.9	7.7
100, on 150	33.1	7.7	88.7	11.3	6.8
150, on 200	18.5	4.3	88.0	12.0	3.8
200	42.8	[a] 10.0	....	....	0.0
	428.5	100.0	....	....	72.2

Total concentrates	per cent	72.2
Slimes	do	10.0
Tailings	do	17.8
		100.0

SAMPLE 9. FRANK WHITE MINE.

Through 20, on 28	113.7	28.4	50.1	49.9	14.2
28, on 35	93.9	23.4	52.5	47.5	12.3
35, on 48	49.1	12.3	65.6	34.4	8.1
48, on 65	27.6	6.9	76.9	23.1	5.3
65, on 100	32.5	8.1	78.6	21.4	6.4
100, on 150	28.4	7.1	80.6	19.4	5.7
150, on 200	18.6	4.6	78.2	21.8	3.6
200	36.9	[a] 9.2	....	....	0.0
	400.7	100.0	....	....	55.6

Total concentrates	per cent	55.6
Slimes	do	9.2
Tailings	do	35.2
		100.0

SAMPLE 10. FRANK WHITE MINE.

Through 20, on 28	51.9	12.7	98.4	1.6	12.5
28, on 35	55.6	13.6	96.5	3.5	13.1
35, on 48	56.2	13.7	92.9	7.1	12.7
48, on 65	40.5	10.0	88.8	11.2	8.9
65, on 100	65.2	15.9	70.0	30.0	11.1
100, on 150	45.5	11.1	76.3	23.7	8.5
150, on 200	23.2	5.7	85.6	14.4	4.9
200	71.0	[a] 17.3	....	....	0.0
	409.1	100.0	....	....	71.7

Total concentrates	per cent	71.7
Slimes	do	17.3
Tailings	do	11.0
		100.0

[a] Slimes.

TABLE 1.—*Results of crushing and separating samples from various mines*—Continued.

SAMPLE 10. FRANK WHITE MINE (UNDER 35 MESH).

Screen analysis.			Separation.		Concentrates.
Size of mesh.	Quantity taken for separation.		Concentrates.	Tailings.	
	Grams.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
Through 35, on 48	26.9	19.1	92.7	7.3	17.7
48, on 65	21.6	15.1	88.9	11.1	13.4
65, on 100	28.2	20.0	69.7	30.3	13.9
100, on 150	20.9	14.8	81.4	18.6	12.0
150, on 200	10.3	7.3	92.3	7.7	6.7
200	33.5	[a] 23.7	....	....	0.0
	141.4	100.0	....	....	63.7

Total concentrates	per cent..	63.7
Slimes	do....	23.7
Tailings	do....	12.6
		100.0

SAMPLE 11. RUFFNER MINE.

Through 20, on 28	37.8	10.2	51.0	49.0	5.2
28, on 35	37.8	10.2	49.2	50.8	5.0
35, on 48	52.7	14.1	49.5	50.5	7.0
48, on 65	68.8	18.5	50.8	49.2	9.4
65, on 100	79.0	21.2	50.2	49.8	10.6
100, on 150	36.3	9.7	59.1	40.9	5.7
150, on 200	17.7	4.7	66.3	33.7	3.1
200	42.6	[a] 11.4	....	....	0.0
	372.7	100.0	....	....	46.0

Total concentrates	per cent..	46.0
Slimes	do....	11.4
Tailings	do....	42.6
		100.0

SAMPLE 11. RUFFNER MINE (UNDER 35 MESH).

Through 35, on 48	35.0	16.5	40.1	59.9	6.6
48, on 65	41.3	19.5	44.1	55.9	8.6
65, on 100	62.3	29.4	42.6	57.4	12.5
100, on 150	25.0	11.8	49.7	50.3	5.9
150, on 200	12.2	5.8	67.3	32.7	3.9
200	36.1	[a] 17.0	....	....	0.0
	211.9	100.0	....	....	37.5

Total concentrates	per cent..	37.5
Slimes	do....	17.0
Tailings	do....	45.5
		100.0

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[a] Slimes.

TABLE 1.—*Results of crushing and separating samples from various mines*—Continued.

SAMPLE 12. SPAULDING MINE.

Screen analysis.			Separation.		Concentrates.
Size of mesh.	Quantity taken for separation.		Concentrates.	Tailings.	
	Grams.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
Through 20, on 28	110.5	26.6	61.8	38.2	16.4
28, on 35	101.7	24.5	66.4	33.6	16.3
35, on 48	65.9	15.9	75.4	24.6	12.0
48, on 65	31.8	7.6	77.5	22.5	5.9
65, on 100	32.5	7.8	78.3	21.7	6.1
100, on 150	26.4	6.4	78.3	21.7	5.0
150, on 200	15.4	3.6	80.4	19.6	2.9
200	31.6	[a]7.6			0.0
	415.8	100.0			64.6

Total concentrates	per cent..	64.6
Slimes	do....	7.6
Tailings	do....	27.8
		100.0

SAMPLE 13. SPAULDING MINE.

Through 20, on 28	118.7	27.4	61.2	38.8	16.8
28, on 35	99.3	22.9	69.0	31.0	15.8
35, on 48	65.5	15.1	73.7	26.3	11.1
48, on 65	35.0	8.0	78.2	21.8	6.3
65, on 100	34.4	7.9	77.1	22.9	6.1
100, on 150	28.2	6.5	79.0	21.0	5.1
150, on 200	17.0	3.9	82.3	17.7	3.2
200	35.8	[a]8.3			0.0
	433.9	100.0			64.4

Total concentrates	per cent..	64.4
Slimes	do....	8.3
Tailings	do....	27.3
		100.0

SAMPLE 14. SPAULDING MINE.

Through 20, on 28	101.0	25.4	61.0	39.0	15.5
28, on 35	88.2	22.2	68.7	31.3	15.3
35, on 48	61.5	15.5	74.0	26.0	11.5
48, on 65	34.7	8.7	75.5	24.5	6.6
65, on 100	34.1	8.5	75.0	25.0	6.4
100, on 150	23.2	5.8	76.0	24.0	4.4
150, on 200	18.8	4.7	76.6	23.4	3.6
200	36.5	[a]9.2			0.0
	398.0	100.0			63.3

Total concentrates	per cent..	63.3
Slimes	do....	9.2
Tailings	do....	27.5
		100.0

[a] Slimes.

TABLE 1.—*Results of crushing and separating samples from various mines*—Continued.

SAMPLE 14. SPAULDING MINE (UNDER 35 MESH).

Screen analysis.			Separation.		Concentrates.
Size of mesh.	Quantity taken for separation.		Concentrates.	Tailings.	
	Grams.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
Through 35, on 48	63.0	28.2	65.1	34.9	18.4
48, on 65	45.0	15.6	68.8	31.2	10.7
65, on 100	36.6	16.4	66.4	33.6	10.9
100, on 150	25.4	11.3	67.5	32.5	7.6
150, on 200	15.8	7.1	71.9	28.1	5.1
200	47.9	[a] 21.4			0.0
	223.7	100.0			52.7

Total concentrates	per cent	52.7
Slimes	do	21.4
Tailings	do	25.9
		100.0

SAMPLE 15. SPAULDING MINE.

Through 20, on 28	112.4	27.7	66.6	33.4	18.5
28, on 35	88.6	21.8	68.4	31.6	14.9
35, on 48	61.9	15.2	75.6	24.4	11.5
48, on 65	28.5	7.0	76.0	24.0	5.3
65, on 100	31.0	7.8	77.4	22.6	6.0
100, on 150	26.1	6.4	77.5	22.5	5.0
150, on 200	17.0	4.2	75.6	24.4	3.2
200	39.1	[a] 9.9			0.0
	405.5	100.0			64.4

Total concentrates	per cent	64.4
Slimes	do	9.9
Tailings	do	25.7
		100.0

SAMPLE 16. SPAULDING MINE.

Through 20, on 28	110.5	24.7	61.7	38.3	15.2
28, on 35	99.0	22.1	70.5	29.5	15.6
35, on 48	67.7	15.1	74.8	25.2	11.3
48, on 65	41.0	9.2	78.9	21.1	7.9
65, on 100	40.5	9.0	77.8	22.2	7.0
100, on 150	27.6	6.2	76.6	23.4	4.7
150, on 200	18.5	4.1	77.2	22.8	3.2
200	43.1	[a] 9.6			0.0
	447.9	100.0			64.9

Total concentrates	per cent	64.9
Slimes	do	9.6
Tailings	do	25.5
		100.0

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[a] Slimes.

TABLE 1.—*Results of crushing and separating samples from various mines*—Continued.

SAMPLE 17. SPAULDING MINE.

Screen analysis.			Separation.		Concentrates.
Size of mesh.	Quantity taken for separation.		Concentrates.	Tailings.	
	Grams.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
Through 20, on 28	110.0	24.2	61.1	38.9	14.4
28, on 35	99.9	22.0	65.5	34.5	14.2
35, on 48	74.2	16.4	74.2	25.8	12.2
48, on 65	36.1	8.0	78.1	21.9	6.4
65, on 100	39.0	8.6	74.6	25.4	6.2
100, on 150	31.8	7.0	73.6	26.4	5.
150, on 200	20.0	4.4	77.5	22.5	3.4
200	42.5	[a] 9.4	....	....	0.0
	453.5	100.0	....	....	62.6

Total concentrates	per cent	62.6
Slimes	do	9.4
Tailings	do	28.0
		100.0

SAMPLE 18. ISHKOODA MINE.

Size of mesh.	Quantity taken for separation (grams).	Quantity taken for separation (per cent).	Concentrates.	Tailings.	Concentrates.
Through 20, on 28	87.1	25.7	67.2	32.8	17.3
28, on 35	77.4	22.8	71.8	28.2	16.4
35, on 48	52.0	15.3	74.5	25.5	11.4
48, on 65	31.6	9.3	75.3	24.7	7.0
65, on 100	27.7	8.2	77.2	22.8	6.3
100, on 150	19.9	5.9	72.6	27.4	4.3
150, on 200	11.8	3.4	73.8	26.2	2.5
200	31.9	[a] 9.4	....	....	0.0
	339.4	100.0	....	....	65.2

Total concentrates	per cent	65.2
Slimes	do	9.4
Tailings	do	25.4
		100.0

SAMPLE 19. ISHKOODA MINE.

Size of mesh.	Quantity taken for separation (grams).	Quantity taken for separation (per cent).	Concentrates.	Tailings.	Concentrates.
Through 20, on 28	100.8	27.2	74.6	25.4	20.3
28, on 35	76.5	20.7	80.6	19.4	16.7
35, on 48	55.9	15.1	81.3	18.7	12.3
48, on 65	31.2	8.4	76.3	23.7	6.4
65, on 100	29.8	8.0	77.0	23.0	6.2
100, on 150	24.2	6.5	80.0	20.0	5.2
150, on 200	14.4	3.9	80.4	19.6	3.1
200	37.8	[a] 10.2	....	....	0.0
	370.6	100.0	....	....	70.2

Total concentrates	per cent	70.2
Slimes	do	10.2
Tailings	do	19.6
		100.0

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[a] Slimes.

TABLE 1.—*Results of crushing and separating samples from various mines*—Continued.

SAMPLE 20. ISHKOODA MINE.

Screen analysis.			Separation.		Concentrates.
Size of mesh.	Quantity taken for separation.		Concentrates.	Tailings.	
	Grams.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
Through 20, on 28	81.5	28.6	57.8	42.2	16.5
28, on 35	67.1	23.6	72.8	27.2	17.2
35, on 48	39.9	14.0	68.9	31.1	9.6
48, on 65	22.0	7.7	69.0	31.0	5.3
65, on 100	22.0	7.7	67.1	32.9	5.2
100, on 150	16.3	5.7	70.3	29.7	4.0
150, on 200	9.5	3.3	80.2	19.8	2.6
200	26.8	[a] 9.4			0.0
	285.1	100.0			60.4

Total concentrates	per cent	60.4
Slimes	do	9.4
Tailings	do	30.2
		100.0

SAMPLE 20. ISHKOODA MINE (UNDER 35 MESH).

Through 35, on 48	65.6	28.2	58.5	41.5	16.5
48, on 65	36.6	15.7	57.9	42.1	9.1
65, on 100	35.7	15.4	60.1	39.9	9.3
100, on 150	29.9	12.9	64.3	35.7	8.3
150, on 200	15.8	6.8	65.1	34.9	4.4
200	48.9	[a] 21.0			0.0
	232.5	100.0			47.6

Total concentrates	per cent	47.6
Slimes	do	21.0
Tailings	do	31.4
		100.0

SAMPLE 21. ISHKOODA MINE.

Through 20, on 28	48.7	19.7	75.4	24.6	14.9
28, on 35	52.1	21.1	80.5	19.5	17.0
35, on 48	40.2	16.3	82.1	17.9	13.4
48, on 65	22.6	9.1	81.5	18.5	7.4
65, on 100	22.0	8.9	79.7	20.3	7.1
100, on 150	19.4	7.8	82.8	17.2	6.5
150, on 200	11.3	4.6	75.8	24.2	3.5
200	30.9	[a] 12.5			0.0
	247.2	100.0			69.8

Total concentrates	per cent	69.8
Slimes	do	12.5
Tailings	do	17.7
		100.0

[a] Slimes.

TABLE 1.—*Results of crushing and separating samples from various mines*—Continued.

SAMPLE 22. ISHKOODA MINE.

Screen analysis.			Separation.		Concentrates.
Size of mesh.	Quantity taken for separation.		Concentrates.	Tailings.	
	Grams.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
Through 20, on 28	120.2	27.5	70.0	30.0	19.3
28, on 35	94.9	21.7	77.1	22.9	16.7
35, on 48	70.6	16.2	81.1	18.9	13.1
48, on 65	42.3	9.7	78.3	21.7	7.6
65, on 100	32.8	7.5	76.7	23.3	5.8
100, on 150	27.7	6.4	77.4	22.6	4.9
150, on 200	14.5	3.3	82.7	17.3	2.7
200	33.5	[a] 7.7			0.0
	436.5	100.0			70.1

Total concentrates	per cent	70.1
Slimes	do	7.7
Tailings	do	22.2
		100.0

SAMPLE 23. SONGO MINE.

Through 20, on 28	90.9	25.4	63.5	36.5	16.1
28, on 35	79.0	22.1	68.9	31.1	15.1
35, on 48	53.9	15.1	72.7	27.3	11.0
48, on 65	30.4	8.5	75.8	24.2	6.5
65, on 100	31.4	8.8	76.8	23.2	6.8
100, on 150	23.8	6.6	76.0	24.0	5.0
150, on 200	12.6	3.5	76.0	24.0	2.6
200	36.0	[a] 10.0			0.0
	358.0	100.0			63.1

Total concentrates	per cent	63.1
Slimes	do	10.0
Tailings	do	26.8
		99.9

SAMPLE 24. SONGO MINE.

Through 20, on 28	95.1	25.4	74.6	25.4	18.9
28, on 35	75.7	20.2	76.7	23.3	15.5
35, on 48	54.9	14.7	78.4	21.6	11.5
48, on 65	33.2	8.9	79.3	20.7	7.1
65, on 100	34.0	9.1	78.2	21.8	7.1
100, on 150	27.7	7.4	75.7	24.3	5.6
150, on 200	12.5	3.3	79.4	20.6	2.6
200	40.8	[a] 11.0			0.0
	373.9	100.0			68.3

Total concentrates	per cent	68.3
Slimes	do	11.0
Tailings	do	20.7
		100.0

[a] Slimes.

TABLE 1.—*Results of crushing and separating samples from various mines*—Continued.

SAMPLE 25. SONGO MINE.

Screen analysis.			Separation.		Concentrates.
Size of mesh.	Quantity taken for separation.		Concentrates.	Tailings.	
	Grams.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
Through 20, on 28	88.4	22.9	72.8	27.2	16.7
28, on 35	77.4	20.0	75.1	24.9	15.0
35, on 48	59.5	15.4	78.0	22.0	12.0
48, on 65	34.4	8.9	77.7	22.3	6.9
65, on 100	35.6	9.2	72.3	27.7	6.6
100, on 150	27.5	7.1	77.2	22.8	5.5
150, on 200	17.3	4.5	75.7	24.3	3.4
200	46.1	[a] 12.0	....	....	0.0
	386.2	100.0	....	....	66.1

Total concentrates	per cent..	66.1
Slimes	do....	12.0
Tailings	do....	21.9
		100.0

SAMPLE 26. SONGO MINE.

Through 20, on 28	96.3	25.3	67.3	32.7	17.0
28, on 35	86.7	22.8	72.5	27.5	16.5
35, on 48	57.5	15.1	74.5	25.5	11.2
48, on 65	31.6	8.3	77.1	22.9	6.4
65, on 100	30.7	8.1	74.5	25.5	6.0
100, on 150	25.3	6.6	74.7	25.3	4.9
150, on 200	13.1	3.4	80.0	20.0	2.8
200	39.6	[a] 10.4	....	....	0.0
	380.8	100.0	....	....	64.8

Total concentrates	per cent..	64.8
Slimes	do....	10.4
Tailings	do....	24.8
		100.0

SAMPLE 27. SONGO MINE.

Through 20, on 28	109.9	26.2	71.6	28.4	18.8
28, on 35	92.5	22.0	75.8	24.2	16.7
35, on 48	63.6	15.1	78.3	21.7	11.8
48, on 65	36.0	8.6	78.3	21.7	6.7
65, on 100	35.8	8.5	78.0	22.0	6.6
100, on 150	25.1	6.0	75.2	24.8	4.5
150, on 200	15.1	3.6	86.0	14.0	3.1
200	42.2	[a] 10.0	....	....	0.0
	420.0	100.0	....	....	68.2

Total concentrates	per cent...	68.2
Slimes	do....	10.0
Tailings	do....	21.8
		100.0

[a] Slimes.

Table 1.—*Results of crushing and separating samples from various mines*—Continued.

SAMPLE 27. SONGO MINE (UNDER 35 MESH).

Screen analysis.			Separation.		Concentrates.
Size of mesh.	Quantity taken for separation.		Concentrates.	Tailings.	
	Grams.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
Through 35, on 48	72.2	27.8	70.2	29.8	19.5
43, on 65	41.8	16.1	71.7	28.3	11.5
65, on 100	42.1	16.2	69.0	31.0	11.2
100, on 150	30.3	11.6	69.7	30.3	8.1
150, on 200	18.7	7.2	76.6	23.4	5.5
200	54.9	[a] 21.1			0.0
	260.0	100.0			55.8

Total concentrates	per cent	55.8
Slimes	do	21.1
Tailings	do	23.1
		100.0

SAMPLE 28. SONGO MINE.

Through 20, on 28	108.2	25.0	71.0	29.0	17.8
28, on 35	93.1	21.5	76.0	24.0	16.3
35, on 48	70.5	16.3	79.9	20.1	13.0
48, on 65	40.5	9.4	80.0	20.0	7.5
65, on 100	35.4	8.2	78.4	21.6	6.4
100, on 150	26.4	6.1	78.6	21.4	4.8
150, on 200	19.0	4.4	80.7	19.3	3.6
200	39.4	[a] 9.1			0.0
	432.5	100.0			69.4

Total concentrates	per cent	69.4
Slimes	do	9.1
Tailings	do	21.5
		100.0

SAMPLE 29. FOSSIL MINE.

Through 20, on 28	93.7	19.0	75.5	24.5	14.3
28, on 35	106.4	21.4	81.8	18.2	17.5
35, on 48	74.9	15.1	80.6	19.4	12.2
48, on 65	49.5	9.9	83.1	16.9	8.2
65, on 100	46.1	9.2	80.3	19.7	7.4
100, on 150	41.2	8.3	83.8	16.2	7.0
150, on 200	29.1	5.8	84.3	15.7	4.9
200	56.2	[a] 11.3			0.0
	497.1	100.0			71.5

Total concentrates	per cent	71.5
Slimes	do	11.3
Tailings	do	17.2
		100.0

---

[a] Slimes.

TABLE 1.—*Results of crushing and separating samples from various mines*—Continued.

SAMPLE 29. FOSSIL MINE (UNDER 35 MESH).

Screen analysis.			Separation.		Concentrates.
Size of mesh.	Quantity taken for separation.		Concentrates.	Tailings.	
	Grams.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
Through 35, on 48	50.4	25.8	77.6	22.4	20.0
48, on 65	31.9	16.4	76.3	23.7	12.5
65, on 100	29.6	15.2	73.0	27.0	11.1
100, on 150	23.5	12.0	74.9	25.1	9.0
150, on 200	15.9	8.1	71.9	28.1	5.8
200	43.8	[a] 22.5			0.0
	195.1	100.0			58.4

Total concentrates	per cent	58.4
Slimes	do	22.5
Tailings	do	19.1
		100.0

SAMPLE 30. FOSSIL MINE.

Through 20, on 28	108.3	24.4	69.0	31.0	16.8
28, on 35	101.5	22.9	75.9	24.1	17.4
35, on 48	70.5	15.9	77.7	22.3	12.4
48, on 65	40.1	9.0	77.5	22.5	7.0
65, on 100	40.0	9.0	74.4	25.6	6.7
100, on 150	29.2	6.6	76.1	23.9	5.0
150, on 200	17.2	3.9	76.6	23.4	3.0
200	36.8	[a] 8.3			0.0
	443.6	100.0			68.3

Total concentrates	per cent	68.3
Slimes	do	8.3
Tailings	do	23.4
		100.0

SAMPLE 31. FOSSIL MINE.

Through 20, on 28	106.3	25.3	67.3	32.7	17.0
28, on 35	92.9	22.1	74.6	25.4	16.5
35, on 48	64.2	15.3	77.6	22.4	11.9
48, on 65	36.5	8.7	77.8	22.2	6.8
65, on 100	36.6	8.7	77.1	22.9	6.7
100, on 150	30.3	7.2	75.3	24.7	5.4
150, on 200	16.0	3.8	74.7	25.3	2.8
200	37.5	[a] 8.9			0.0
	420.3	100.0			67.1

Total concentrates	per cent	67.1
Slimes	do	8.9
Tailings	do	24.0
		100.0

[a] Slimes.

TABLE 1.—*Results of crushing and separating samples from various mines*—Continued.

SAMPLE 32. FOSSIL MINE.

Screen analysis.			Separation.		Concentrates.
Size of mesh.	Quantity taken for separation.		Concentrates.	Tailings.	
	Grams.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
Through 20, on 28	109.5	27.4	77.0	23.0	21.1
28, on 35	80.0	20.0	81.8	18.2	16.4
35, on 48	54.7	13.7	78.4	21.6	10.7
48, on 65	33.8	8.5	77.7	22.3	6.6
65, on 100	32.8	8.2	76.2	23.8	6.2
100, on 150	30.4	7.6	75.6	24.4	5.7
150, on 200	19.7	4.9	78.1	21.9	3.8
200	38.6	[a] 9.7	....	....	0.0
	399.5	100.0	....	....	70.5

Total concentrates	per cent	70.5
Slimes	do	9.7
Tailings	do	19.8
		100.0

SAMPLE 33. FOSSIL MINE.

Through 20, on 28	96.8	25.7	69.8	30.2	17.9
28, on 35	82.6	21.9	78.1	21.9	17.1
35, on 48	58.5	15.5	80.7	19.3	12.5
48, on 65	34.6	9.2	80.8	19.3	7.4
65, on 100	30.5	8.1	79.1	20.9	6.4
100, on 150	25.5	6.7	79.6	20.4	5.3
150, on 200	14.2	3.7	77.3	22.7	2.9
200	34.8	[a] 9.2	....	....	0.0
	377.5	100.0	....	....	69.5

Total concentrates	per cent	69.5
Slimes	do	9.2
Tailings	do	21.3
		100.0

SAMPLE 34. FOSSIL MINE.

Through 20, on 28	106.2	25.8	63.8	36.2	16.5
28, on 35	88.4	21.4	72.7	27.3	15.6
35, on 48	64.3	15.6	75.4	24.6	11.8
48, on 65	36.2	8.8	73.9	26.1	6.5
65, on 100	33.9	8.2	73.7	26.3	6.0
100, on 150	26.9	6.5	74.2	25.8	4.8
150, on 200	16.9	4.1	75.9	24.1	3.1
200	39.7	[a] 9.6	....	....	0.0
	412.5	100.0	....	....	64.3

Total concentrates	per cent	64.3
Slimes	do	9.6
Tailings	do	26.1
		100.0

[a] Slimes.

TABLE 1.—*Results of crushing and separating samples from various mines*—Continued.

SAMPLE 35. RUFFNER MINE.

Screen analysis.			Separation.		Concentrates.
Size of mesh.	Quantity taken for separation.		Concentrates.	Tailings.	
	Grams.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
Through 20, on 28	129.3	27.1	70.9	29.1	19.2
28, on 35	113.4	23.8	78.5	21.5	18.7
35, on 48	78.5	16.5	78.2	21.8	12.9
48, on 65	35.6	7.5	77.6	22.4	5.8
65, on 100	37.7	7.9	77.0	23.0	6.1
100, on 150	25.6	5.4	76.7	23.3	4.1
150, on 200	19.5	4.0	80.0	20.0	3.2
200	37.4	[a]7.8	....	....	0.0
	477.0	100.0	....	....	70.0

Total concentrates	per cent	70.0
Slimes	do	7.8
Tailings	do	22.2
		100.0

SAMPLE 36. RUFFNER MINE.

Through 20, on 28	95.2	25.3	66.4	33.6	16.8
28, on 35	79.2	21.0	74.4	25.6	15.6
35, on 48	53.0	14.1	74.7	25.3	10.5
48, on 65	34.5	9.2	72.0	28.0	6.6
65, on 100	34.7	9.2	70.7	29.3	6.5
100, on 150	25.9	6.9	70.5	29.5	4.9
150, on 200	16.7	4.4	70.0	30.0	3.1
200	37.1	[a]9.9	....	....	0.0
	376.3	100.0	....	....	64.0

Total concentrates	per cent	64.0
Slimes	do	9.9
Tailings	do	26.1
		100.0

SAMPLE 37. HAMMOND MINE.

Through 20, on 28	103.6	25.8	74.5	25.5	19.2
28, on 35	92.4	23.0	83.8	16.2	19.3
35, on 48	61.7	15.3	80.4	19.6	12.3
48, on 65	32.3	8.0	80.5	19.5	6.4
65, on 100	32.6	8.1	82.6	17.4	6.7
100, on 150	27.4	6.8	84.7	15.3	5.8
150, on 200	14.1	3.5	91.4	8.6	3.2
200	38.2	[a]9.5	....	....	0.0
	402.3	100.0	....	....	72.9

Total concentrates	per cent	72.9
Slimes	do	9.5
Tailings	do	17.6
		100.0

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[a] Slimes.

TABLE 1.—*Results of crushing and separating samples from various mines*—Continued.

SAMPLE 38. RUFFNER MINE.

Screen analysis.			Separation.		Concentrates.
Size of mesh.	Quantity taken for separation.		Concentrates.	Tailings.	
	Grams.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
Through 20, on 28	103.9	29.7	39.2	60.8	11.6
28, on 35	86.1	24.6	42.8	57.2	10.5
35, on 48	48.4	13.8	51.6	48.4	7.1
48, on 65	24.6	7.0	60.3	39.7	4.2
65, on 100	25.9	7.0	65.4	34.6	4.8
100, on 150	20.2	5.8	63.7	36.3	3.7
150, on 200	11.8	3.4	66.6	33.4	2.3
200	29.1	[a] 8.3	....	....	0.0
	350.0	99.6	....	....	44.2

Total concentrates	per cent	44.2
Slimes	do	8.3
Tailings	do	47.5
		100.0

SAMPLE 39. HAMMOND MINE.

Size of mesh.	Grams.	Per cent.	Concentrates.	Tailings.	Concentrates.
Through 20, on 28	81.3	20.7	74.8	25.2	15.5
28, on 35	88.1	22.4	66.7	33.3	14.9
35, on 48	60.1	15.3	61.0	39.0	9.3
48, on 65	33.2	8.7	86.5	13.5	7.5
65, on 100	39.7	10.1	89.6	10.4	9.0
100, on 150	30.6	7.7	91.3	8.7	7.0
150, on 200	16.4	4.1	90.4	9.6	3.7
200	43.6	[a] 11.0	....	....	0.0
	393.0	100.0	....	....	66.9

Total concentrates	per cent	66.9
Slimes	do	11.0
Tailings	do	22.1
		100.0

[a] Slimes.

TABLE 2.—*Composition of products and recoveries of samples from various mines.*

SAMPLE 1. ISKOODA MINE.

Products.	Fe.	SiO_2.	CaO.	Total iron.	Total silica.	Ratio of concentration.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	
Crude	32.1	10.3	21.1	100.0	100.0	....
Concentrates	43.5	7.0	12.6	80.7	38.2	1.74
Slimes	33.9	7.3	....	10.5	6.7	....
Tailings	8.2	17.6	....	8.8	55.1	....
Concentrates+slimes	42.1	7.0	....	91.2	44.9	1.49

SAMPLE 2. SPAULDING MINE.

Products.	Fe.	SiO_2.	CaO.	Total iron.	Total silica.	Ratio of concentration.
Crude	36.9	20.5	11.1	100.0	100.0	....
Concentrates	46.3	10.9	18.5	85.6	34.4	1.46
Slimes	44.8	12.1	....	8.2	3.8	....
Tailings	9.2	49.6	....	6.2	61.8	....
Concentrates+slimes	46.1	11.0	....	93.8	38.2	1.33

TABLE 2.—*Composition of products and recoveries of samples from various mines*—Continued.

SAMPLE 3. SPAULDING MINE.

Products.	Fe.	SiO₂.	CaO.	Total iron.	Total silica.	Ratio of concentration.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	
Crude	35.7	15.0	....	100.0	100.0	....
Concentrates	46.2	7.9	....	83.8	34.3	1.54
Slimes	40.0	8.9	....	10.3	5.5	....
Tailings	8.2	34.7	....	5.9	60.2	....
Concentrates+slimes	45.4	8.0	....	94.1	39.8	1.35

SAMPLE 4. HAMMOND MINE.

Crude	37.2	40.3	....	100.0	100.0	....
Concentrates	52.4	17.5	....	80.7	21.5	1.85
Slimes	47.1	23.9	....	12.1	4.9	....
Tailings	6.8	87.2	....	7.2	73.6	....
Concentrates+slimes	51.7	18.4	....	92.8	26.4	1.59

SAMPLE 5. RUFFNER MINE.

Crude	28.8	31.8	10.2	100.0	100.0	....
Concentrates	40.2	20.2	6.9	71.2	30.2	2.07
Slimes	40.4	11.6	....	16.4	4.0	....
Tailings	8.7	55.1	....	12.4	65.8	....
Concentrates+slimes	40.3	18.6	....	87.6	34.2	1.65

SAMPLE 6. RUFFNER MINE.

Crude	28.8	29.9	....	100.0	100.0	....
Concentrates	43.7	16.7	....	64.0	22.3	2.44
Slimes	34.5	13.0	....	24.4	8.4	....
Tailings	8.3	54.4	....	11.6	69.3	....
Concentrates+slimes	40.7	15.5	....	88.4	30.7	1.65

SAMPLE 7. FRANK WHITE MINE.

Crude	45.2	26.1	....	100.0	100.0	....
Concentrates	53.6	13.4	....	84.1	32.8	1.45
Slimes	53.7	12.7	....	11.5	4.2	....
Tailings	8.9	81.2	....	4.4	63.0	....
Concentrates+slimes	53.6	13.3	....	95.6	37.0	1.28

SAMPLE 8. FRANK WHITE MINE.

Crude	44.0	24.5	....	100.0	100.0	....
Concentrates	52.3	12.7	....	84.9	37.8	1.39
Slimes	53.0	11.3	....	11.9	4.6	....
Tailings	8.0	78.6	....	3.2	57.6	....
Concentrates+slimes	52.4	12.5	....	96.8	42.4	1.22

SAMPLE 9. FRANK WHITE MINE.

Crude	36.3	41.0	....	100.0	100.0	....
Concentrates	49.8	19.7	....	80.3	25.1	1.80
Slimes	46.4	23.4	....	12.4	4.9	....
Tailings	7.2	86.9	....	7.3	70.0	....
Concentrates+slimes	49.3	20.2	....	92.7	30.0	1.54

TABLE 2.—*Composition of products and recoveries of samples from various mines*—Continued.

SAMPLE 10. FRANK WHITE MINE.

Products.	Fe.	SiO_2.	CaO.	Total iron.	Total silica.	Ratio of concentration.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	
Crude	45.8	25.1	....	100.0	100.0	....
Concentrates	49.8	19.1	....	69.7	47.2	1.57
Slimes	53.7	12.7	....	28.0	11.7	....
Tailings	8.4	84.3	....	2.3	41.1	....
Concentrates+slimes	50.9	17.4	....	97.7	58.9	1.14

SAMPLE 11. RUFFNER MINE.

Crude	27.7	34.3	10.1	100.0	100.0	....
Concentrates	44.0	17.7	4.7	61.0	18.3	2.67
Slimes	39.1	11.7	....	24.6	5.5	....
Tailings	8.6	60.7	....	14.4	76.2	....
Concentrates+slimes	42.7	15.8	....	85.6	23.8	1.83

SAMPLE 12. SPAULDING MINE.

Crude	35.9	27.0	....	100.0	100.0	....
Concentrates	45.5	11.9	....	83.1	27.7	1.55
Slimes	46.1	14.0	....	9.9	3.8	....
Tailings	8.9	68.4	....	7.0	68.5	....
Concentrates+slimes	45.6	12.1	....	93.0	31.5	1.39

SAMPLE 13. SPAULDING MINE.

Crude	36.8	26.2	7.5	100.0	100.0	....
Concentrates	45.6	13.2	7.6	82.1	30.8	1.55
Slimes	46.4	13.1	....	10.8	3.9	....
Tailings	9.3	66.0	....	7.1	65.3	....
Concentrates+slimes	45.7	13.2	....	92.9	34.7	1.38

SAMPLE 14. SPAULDING MINE.

Crude	35.3	27.7	....	100.0	100.0	....
Concentrates	45.1	13.5	....	81.4	30.4	1.58
Slimes	44.3	14.9	....	11.6	4.8	....
Tailings	8.7	66.3	....	7.0	64.8	....
Concentrates+slimes	44.9	13.6	....	93.0	35.2	1.38

SAMPLE 15. SPAULDING MINE.

Crude	36.5	26.1	....	100.0	100.0	....
Concentrates	45.0	14.0	....	81.4	32.8	1.55
Slimes	44.4	14.4	....	12.3	5.2	....
Tailings	8.7	66.6	....	6.3	62.0	....
Concentrates+slimes	44.9	14.1	....	93.7	38.0	1.35

SAMPLE 16. SPAULDING MINE.

Crude	36.9	26.1	....	100.0	100.0	....
Concentrates	44.9	13.1	....	81.2	31.1	1.54
Slimes	46.5	13.5	....	12.4	4.4	....
Tailings	9.0	69.1	....	6.4	64.5	....
Concentrates+slimes	45.1	13.0	....	93.6	35.5	1.34

TABLE 2.—*Composition of products and recoveries of samples from various mines*—Continued.

SAMPLE 17, SPAULDING MINE.

Products.	Fe.	SiO_2.	CaO.	Total iron.	Total silica.	Ratio of concentration.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	
Crude	35.0	27.5		100.0	100.0	
Concentrates	47.7	12.3		82.3	27.8	1.60
Slimes	43.3	15.4		11.2	5.2	
Tailings	8.4	66.4		6.5	67.0	
Concentrates and slimes	47.1	12.7		93.5	33.0	1.39
AVERAGE OF SAMPLES 12 TO 17.						
Crude	36.1	26.8	7.5			
Concentrates	45.6	13.0	7.6	81.9		1.56
Slimes	45.2	14.2				
Tailings	8.8	67.1				
Concentrates and slimes	45.6	13.1		93.3		1.37
SAMPLE 18. ISHKOODA MINE.						
Crude	33.5	29.7	8.0	100.0	100.0	
Concentrates	43.7	14.2	7.7	80.6	33.9	1.55
Slimes	45.4	13.2		12.1	4.5	
Tailings	10.2	66.5		7.3	61.6	
Concentrates and slimes	43.9	14.0		92.7	38.4	1.34
SAMPLE 19. ISHKOODA MINE.						
Crude	39.1	20.5		100.0	100.0	
Concentrates	47.1	10.6		83.5	39.8	1.42
Slimes	45.3	10.6		11.6	5.8	
Tailings	9.8	52.0		4.9	54.4	
Concentrates and slimes	46.9	10.6		95.1	45.6	1.24
SAMPLE 20. ISHKOODA MINE.						
Crude	32.4	29.3		100.0	100.0	
Concentrates	42.3	16.1		80.0	34.5	1.66
Slimes	39.8	12.4		11.7	4.1	
Tailings	8.8	57.3		8.3	61.4	
Concentrates and slimes	42.0	15.6		91.7	38.6	1.44
SAMPLE 21. ISHKOODA MINE.						
Crude	42.3	18.2		100.0	100.0	
Concentrates	48.9	9.6		81.4	37.6	1.44
Slimes	49.0	11.2		14.6	7.8	
Tailings	9.4	55.0		4.0	54.6	
Concentrates and slimes	48.9	9.8		96.0	45.4	1.21
SAMPLE 22. ISHKOODA MINE.						
Crude	38.8	20.2		100.0	100.0	
Concentrates	46.2	11.3		85.9	37.7	1.43
Slimes	45.0	11.6		9.2	4.2	
Tailings	8.4	55.0		4.9	58.1	
Concentrates and slimes	46.1	11.3		95.1	41.9	1.30

TABLE 2.—*Composition of products and recoveries of samples from various mines*—Continued.

AVERAGE OF SAMPLES 18 TO 22.

Products.	Fe.	SiO_2.	CaO.	Total iron.	Total silica.	Ratio of concentration.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	
Crude	37.1	25.6	8.0			
Concentrates	45.6	12.4	7.7	82.3		1.50
Slimes	44.9	11.8				
Tailings	9.3	57.2				
Concentrates and slimes	45.6	12.3		94.1		1.31

SAMPLE 23. SONGO MINE.

Crude	37.1	21.8		100.0	100.0	
Concentrates	45.4	12.7		81.1	32.0	1.58
Slimes	43.9	12.9		12.4	5.1	
Tailings	8.6	58.8		6.5	62.9	
Concentrates+slimes	45.2	12.7		93.5	37.1	1.37

SAMPLE 24. SONGO MINE.

Crude	38.2	19.7		100.0	100.0	
Concentrates	45.3	12.3		82.5	40.4	1.46
Slimes	44.1	11.2		12.9	5.9	
Tailings	8.4	53.9		4.6	53.7	
Concentrates+slimes	45.1	12.1		95.4	46.3	1.26

SAMPLE 25. SONGO MINE.

Crude	39.1	21.9		100.0	100.0	
Concentrates	46.2	12.6		78.1	37.9	1.51
Slimes	45.8	14.1		14.0	7.7	
Tailings	14.1	54.4		7.9	54.4	
Concentrates+slimes	46.1	12.8		92.1	45.6	1.28

SAMPLE 26. SONGO MINE.

Crude	39.3	23.2		100.0	100.0	
Concentrates	44.9	12.4		81.1	33.6	1.54
Slimes	43.7	13.5		12.7	5.8	
Tailings	9.0	58.6		6.2	60.6	
Concentrates+slimes	44.7	12.6		93.8	39.4	1.33

SAMPLE 27. SONGO MINE.

Crude	37.2	21.1	10.5	100.0	100.0	
Concentrates	43.5	13.2	9.5	83.2	37.5	1.47
Slimes	44.0	12.8		12.3	5.3	
Tailings	7.4	63.0		4.5	57.2	
Concentrates+slimes	43.6	13.1		95.5	42.8	1.28

SAMPLE 28. SONGO MINE.

Crude	39.5	19.3		100.0	100.0	
Concentrates	45.3	11.8		83.9	38.0	1.44
Slimes	47.4	11.7		11.5	4.9	
Tailings	8.0	57.2		4.6	57.1	
Concentrates+slimes	45.5	11.8		95.4	42.9	1.27

TABLE 2.—*Composition of products and recoveries of samples from various mines*—Continued.

AVERAGE OF SAMPLES 23 TO 28.

Products.	Fe.	SiO_2.	CaO.	Total iron.	Total silica.	Ratio of concentration.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	
Crude	38.4	21.2	10.5			
Concentrates	45.1	12.5	9.5	81.7		1.50
Slimes	44.8	12.7				
Tailings	9.3	57.7				
Concentrates+slimes	45.0	12.5		94.3		1.30
SAMPLE 29. FOSSIL MINE.						
Crude	39.8	21.1		100.0	100.0	
Concentrates	48.1	11.7		71.1	31.8	1.71
Slimes	43.8	16.6		25.0	17.4	
Tailings	8.1	57.1		3.9	50.8	
Concentrates+slimes	46.9	13.1		96.1	49.2	1.24
SAMPLE 30. FOSSIL MINE.						
Crude	37.8	22.0		100.0	100.0	
Concentrates	46.0	11.9		84.5	36.2	1.46
Slimes	44.6	13.6		9.9	5.0	
Tailings	8.9	56.3		5.6	58.8	
Concentrates+slimes	45.8	12.1		94.4	41.2	1.31
SAMPLE 31. FOSSIL MINE.						
Crude	36.8	21.7	9.6	100.0	100.0	
Concentrates	45.3	12.8	7.6	84.0	38.8	1.49
Slimes	43.0	13.7		10.6	5.5	
Tailings	8.2	51.4		5.4	55.7	
Concentrates+slimes	45.0	12.9		94.6	44.3	1.32
SAMPLE 32. FOSSIL MINE.						
Crude	39.4	18.6		100.0	100.0	
Concentrates	46.2	11.9		84.7	42.4	1.42
Slimes	44.2	12.4		11.1	6.1	
Tailings	8.3	51.5		4.2	51.5	
Concentrates+slimes	46.0	12.0		95.8	48.5	1.35
SAMPLE 33. FOSSIL MINE.						
Crude	38.4	20.2		100.0	100.0	
Concentrates	45.3	12.0		84.0	38.5	1.44
Slimes	45.5	11.3		11.2	4.8	
Tailings	8.5	57.5		4.8	56.7	
Concentrates+slimes	45.3	11.9		95.2	43.3	1.27
SAMPLE 34. FOSSIL MINE.						
Crude	35.6	21.5		100.0	100.0	
Concentrates	44.9	12.2		82.4	35.3	1.56
Slimes	42.0	12.9		11.5	5.6	
Tailings	8.2	50.4		6.1	59.1	
Concentrates+slimes	44.5	12.3		93.9	38.9	1.35

TABLE 2.—*Composition of products and recoveries of samples from various mines*—Continued.

AVERAGE OF SAMPLES 30 TO 34.

Products.	Fe.	SiO_2.	CaO.	Total iron.	Total silica.	Ratio of concentration.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	
Crude	37.6	20.8	9.6	....	....	....
Concentrates	45.5	12.2	7.6	83.8	....	1.47
Slimes	43.9	12.8	....	....	....	....
Tailings	8.4	53.3	....	....	....	....
Concentrates+slimes	45.3	12.2	....	94.8	....	1.30
SAMPLE 35. RUFFNER MINE.						
Crude	38.1	22.1	....	100.0	100.0	....
Concentrates	45.1	13.7	....	85.4	40.4	1.43
Slimes	44.3	14.2	....	9.4	4.7	....
Tailings	8.6	58.6	....	5.2	54.9	....
Concentrates + slimes	44.9	13.8	....	94.8	45.1	1.29
SAMPLE 36. RUFFNER MINE.						
Crude	34.5	23.2	....	100.0	100.0	....
Concentrates	43.5	15.0	....	82.8	40.0	1.56
Slimes	40.4	13.3	....	11.9	5.5	....
Tailings	6.9	50.1	....	5.3	54.5	....
Concentrates + slimes	43.1	14.7	....	94.7	45.5	1.35
SAMPLE 37. HAMMOND MINE.						
Crude	41.5	20.9	....	100.0	100.0	....
Concentrates	46.8	14.0	....	84.6	45.3	1.37
Slimes	50.0	11.1	....	11.8	4.7	....
Tailings	8.4	64.1	....	3.6	50.0	....
Concentrates + slimes	47.2	13.7	....	96.4	50.0	1.21
SAMPLE 38. RUFFNER MINE.						
Crude	28.2	40.8	....	100.0	100.0	....
Concentrates	47.5	14.7	....	77.8	14.5	2.26
Slimes	38.9	23.8	....	12.0	4.4	....
Tailings	5.8	76.6	....	10.2	81.1	....
Concentrates + slimes	46.1	16.1	....	89.8	18.9	1.90
SAMPLE 39. HAMMOND MINE.						
Crude	46.2	28.0	....	100.0	100.0	....
Concentrates	55.2	14.6	....	82.9	32.1	1.50
Slimes	55.5	13.3	....	13.7	4.8	....
Tailings	6.8	87.1	....	3.4	63.1	....
Concentrates + slimes	55.3	14.4	....	96.6	36.9	1.28

NOTES ON SAMPLES.

ORE FROM THE BIG SEAM.

SAMPLE 1. ISHKOODA MINE.

Sample 1 was taken in No. 13 slope, 6 right heading, near the manway. The upper bench is here underlaid with a ½-inch shale parting and is 7 feet thick. The sample was taken across the entire bench.

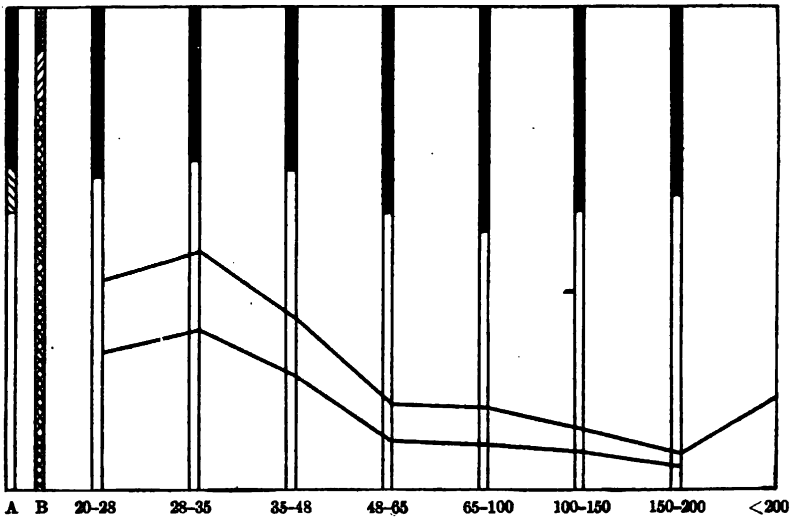

FIGURE 1.—Results of tests of sample 1.[a]

SAMPLE 2. SPAULDING MINE.

Sample 2, from 1 left heading, cross left, 700 feet from slope. The Big seam has no parting in this mine, so that the lower limit of the upper bench is an arbitrary line, and the upper 11 feet of the seam were mined when the mine was working. The sample represents the entire 11 feet of workable ore.

SAMPLE 3. SPAULDING MINE.

Sample 3 was taken from 3 left heading at the breast and represents the upper 12 feet of the seam or the part that was worked at this point.

Instead of the concentrates and tailings for the entire sample being analyzed, the concentrates and tailings for each mesh size were analyzed, and the composition of the concentrates and tailings, as given above, was calculated from these results. The analyses of the products obtained from the different mesh sizes follow. For each mesh size the percentage of the total iron that went into the concentrates and the tailings in that size is given, and also the composition of the crude ore of each size.

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[a] In figs. 1 to 47, column A shows, in upward order, the percentage of concentrate, slime, and tailing from the sample, and column B the percentage in each of the total iron in the sample. Upper broken curve shows proportion of total crushed ore in each size; lower broken curve shows actual quantity of concentrate from each size. See p. 15.

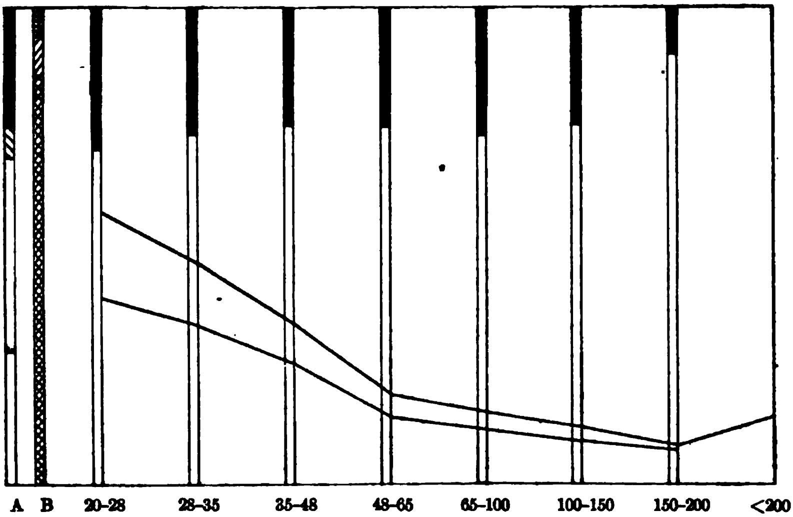

FIGURE 2.—Results of tests of sample 2.

Composition of concentrates and tailings by mesh sizes.

Size of mesh.	Concentrates.		Tailings.		Crude.		Total iron in—	
	Fe.	SiO_2.	Fe.	SiO_2.	Fe.	SiO_2.	Concentrates.	Tailings.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
20 to 28	42.9	9.5	9.3	44.7	32.5	20.5	91.1	8.9
28 to 35	45.7	8.1	10.0	36.9	36.1	15.9	92.6	7.4
35 to 48	46.4	7.6	8.9	33.5	37.1	14.0	94.0	6.0
48 to 65	46.8	6.9	8.1	25.0	35.0	12.4	93.0	7.0
65 to 100	47.5	6.9	6.4	24.9	35.3	12.2	94.7	5.3
100 to 150	51.7	6.1	6.6	22.5	38.4	10.9	95.0	5.0
150 to 200	55.1	5.7	9.0	20.0	42.3	9.7	94.1	5.9
200	....	....	....	....	40.0	8.9	....	....

The above table shows that the 20 to 28 mesh crude ore is of decidedly lower grade than the original ore, the 28 to 35 mesh size is of about the same grade, and the smaller sizes are all better than the original, there being a nearly constant increase in grade for each successively smaller size. If at the outset of concentration the 20 to 28 mesh size were rejected and thrown into the tailings, with a ratio of concentration of 2.11 and and an iron recovery of 62.8 per cent, one would obtain a concentrate running 47.4 per cent Fe and 7.3

per cent SiO_2. In other words, the compensation for a decrease in iron recovery of 21 per cent would be by only an increase of 1.2 per

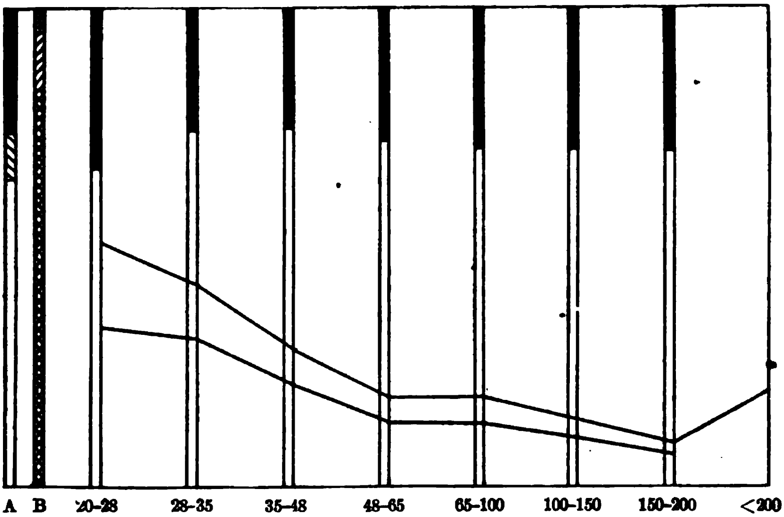

Figure 3.—Results of tests of sample 3.

cent iron and a decrease of 0.6 per cent silica in the concentrates, not enough to warrant the procedure.

SAMPLE 4. HAMMOND MINE.

The Big seam was not worked at this mine, but it is cut by an adit driven to reach the slope on the Irondale seam, and the sample was

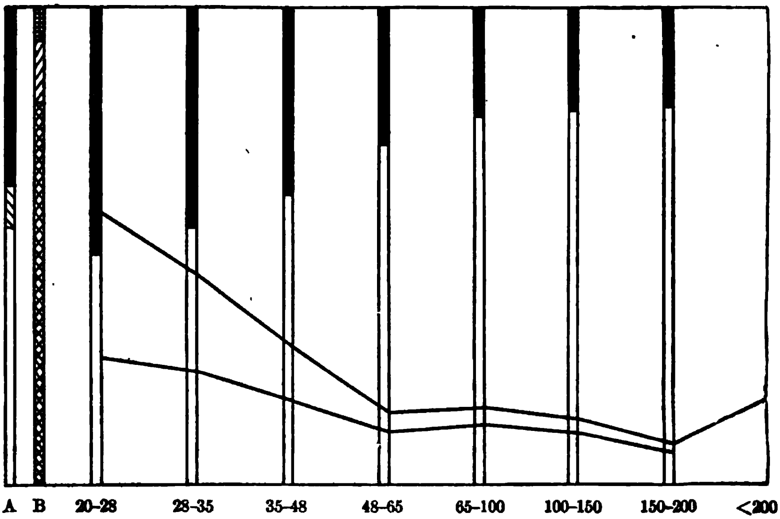

Figure 4.—Results of tests of sample 4.

taken from the exposure in this adit. The seam here is only a few feet from the surface, and the ore corresponds more nearly to soft ore. The sample was taken across the entire seam.

SAMPLE 5. RUFFNER MINE.

The Big seam in the Ruffner mine is about 18 feet thick, the upper 7 feet being workable, and is overlain by what is called "honeycomb" rock, which is really a low-grade ore high in silica with considerable visible calcite. The sample was taken from the 7 feet of workable ore at the bottom of the slope at heading 10 left. The ore is unusually fine grained, as is shown by the screen-analysis curves (figs. 5 to 8).

Because the ore was so fine grained another part of the sample was crushed to pass 35 mesh and subjected to separation. The results of this test, given in Table 1 (p. 19), show a screen-analysis curve (fig. 6) of the same character as that from the 20-mesh test and the same percentage of tailings in the concentration tests. The only evident change is an increase of slimes and a decrease in the quantity of concentrates with finer crushing. Though no analysis was made of the products, the various percentages seem to indicate that little was gained by the finer crushing.

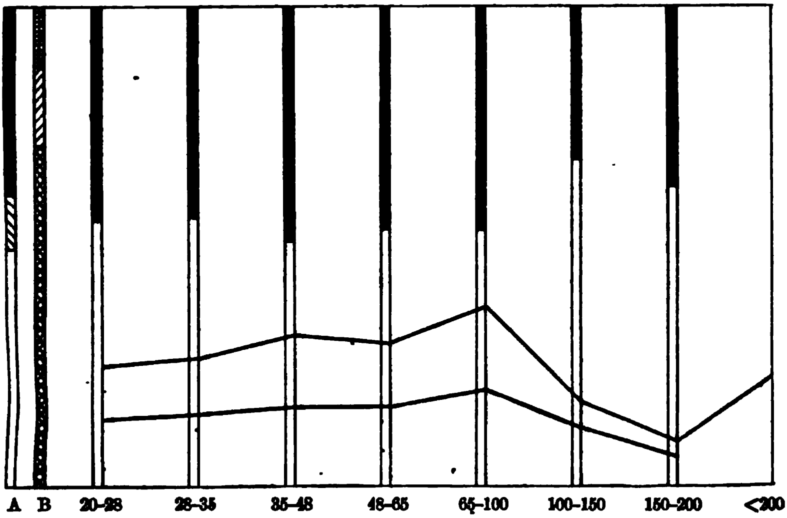

FIGURE 5.—Results of tests of sample 5.

SAMPLE 6. RUFFNER MINE.

Sample 6 was taken from 4 left heading, 400 feet from the slope, and represents the upper 7 feet of ore. This ore is very fine grained, as the screen-analysis curves indicate. Only the concentrates from this separation were analyzed. They carried 42.5 per cent Fe and 20.6 per cent SiO_2. Another part of this sample was crushed under 35 mesh and concentrated; the results are given on page 20.

Crushing this sample finer than 35 mesh increased the ratio of concentration from 1.64 to 2.44, and correspondingly increased the grade of the concentrates. The increase of slimes leaves the advantage still decidedly in favor of the 35-mesh size, for if the slimes are added to the concentrates the ratio of concentration becomes 1.65, which is practically that of the concentrates in the 20-mesh size, and yet the

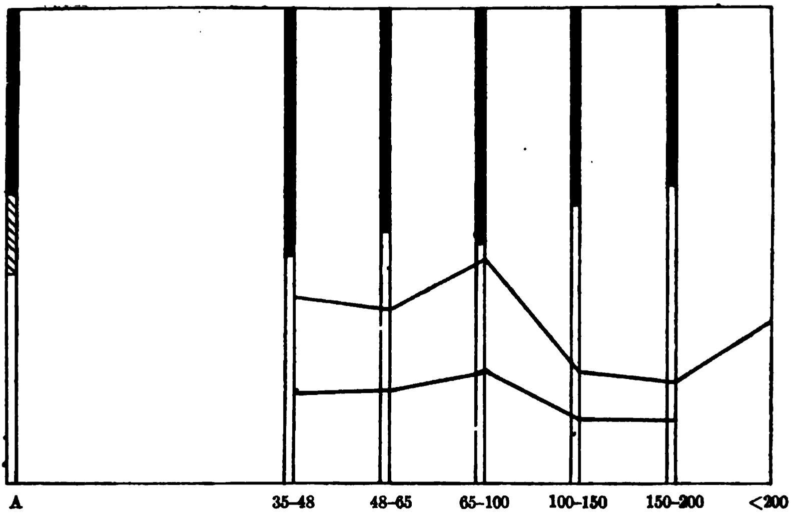

FIGURE 6.—Results of tests of sample 5 under 35 mesh.

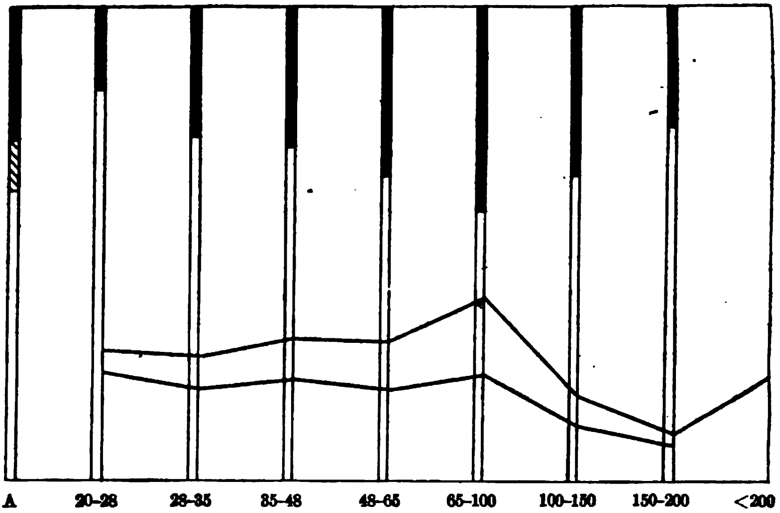

FIGURE 7.—Results of tests of sample 6.

composition of the combined product runs only 1.5 per cent less in iron and 5 per cent less in silica. If the slimes are rejected and only the concentrates considered, the finer crushing yields a product 1.2 per cent higher in iron and 3.9 per cent lower in silica, but decreases the iron recovery to 64 per cent.

COMMENTS ON TESTS OF UPPER BENCH ORE.

Of the preceding six samples of Big seam ore the Hammond sample represents soft ore from the entire thickness of the seam, the Ishkooda sample is self-fluxing, and the other four are siliceous hard ores. The two from the Spaulding mine are comparatively good for ores of that type, but the two from the Ruffner mine are low in iron and high in silica and can not be utilized in their present condition.

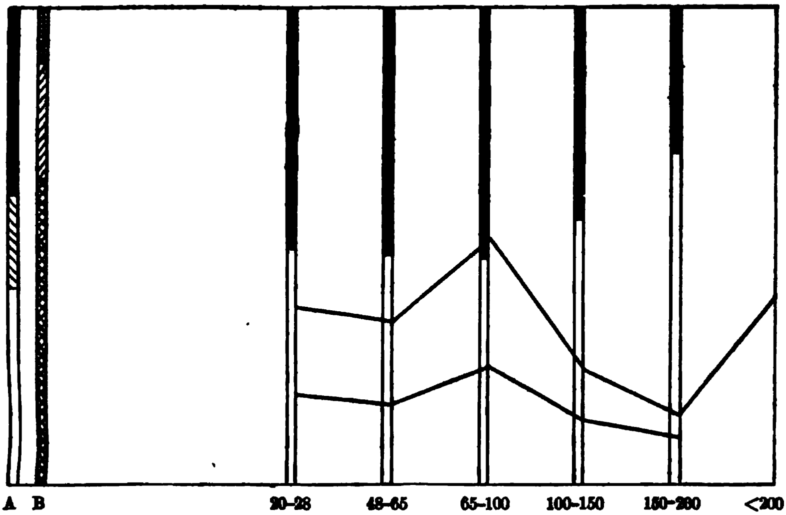

FIGURE 8.—Results of tests of sample 6 under 35 mesh.

Concentration of the self-fluxing Ishkooda ore yielded a self-fluxing concentrate more than 10 per cent higher in iron and 3 per cent lower in silica than the original ore. Though the product is more valuable, concentration would hardly be justified because the crude ore is itself marketable.

The Spaulding ores represent those that are too siliceous to work except when the iron market is strong. The average of the two samples is 36.3 per cent Fe and 17.8 per cent SiO_2, and in one sample the lime content is 11.1. The concentrates average 46.3 per cent Fe and 9.4 per cent SiO_2, and the lime content of one sample is 8.5. In other words, concentration of a decidedly siliceous ore yielded a concentrate carrying 10 per cent more iron, 8.4 per cent less silica, and nearly self-fluxing. Addition of the slimes to the concentrates makes a product averaging 45.8 per cent Fe and 9.5 per cent SiO_2, a reduction of only 0.5 per cent Fe and an increase of 0.1 SiO_2, with an increase of iron recovery from 84.7 to 94 per cent. Thus the concentration of the upper-bench Spaulding ore, which has been utilized only when the iron trade was most active, yielded a product better than the best of the crude ores of the district.

The soft Hammond ore, which in its crude state is too low in iron and high in silica to have any value, yielded a concentrate somewhat better than the average soft ore of the district, which according to Phillips[a] carries 50.80 per cent Fe and 18.50 per cent SiO_2.

Least promising are the results from the two samples of Ruffner ore. They average 28.8 per cent Fe and 30.9 per cent SiO_2, and the concentrates average 42 per cent Fe and 18.5 per cent SiO_2. In one sample the lime content fell from 10.2 in the crude ore to 6.9 per cent in the concentrates. The average of concentrates plus slimes is 40.5 per cent Fe and 17.1 per cent SiO_2, and the iron recovery is thereby increased from 67.6 per cent to 88 per cent. Either product is below the limits of marketable ore.

BIG SEAM ORE FROM FRANK WHITE MINE.

Four samples of soft ore were taken from the open cut of the Frank White mine, on the summit of Red Mountain, at the south end of Birmingham. An incline leads from this open cut, which is several hundred feet long, to the railroad tracks. At the top of the incline the entire thickness, 18 feet, of the Big seam is exposed. Only the upper 6 feet has been worked, and from it two samples were taken, one from each side of the incline. A third sample was taken at the incline, from the middle 6 feet of the bed, and a fourth at the same place from the lowest 6 feet, which is much finer grained than the rest of the bed, as indicated by the screen-analysis curves (figs. 12 and 13). The ore from the middle 6 feet is similar in appearance to that from the upper, but, as the analyses show, is much poorer, being high in silica and low in iron for a soft ore. Because of the three divisions of the bed, and the samples being taken at the outcrop, they are considered apart from the rest of the Big seam samples.

SAMPLE 7. FRANK WHITE MINE.

Sample 7 represents the upper 6 feet of the Big seam in the part of the open cut south of the incline.

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[a] Phillips, W. B., Iron making in Alabama, 3d ed.: Alabama Geological Survey, 1912, p. 80.

SAMPLE 8. FRANK WHITE MINE.

Sample 8 represents the upper 6 feet of the Big seam in the part of the open cut north of the incline.

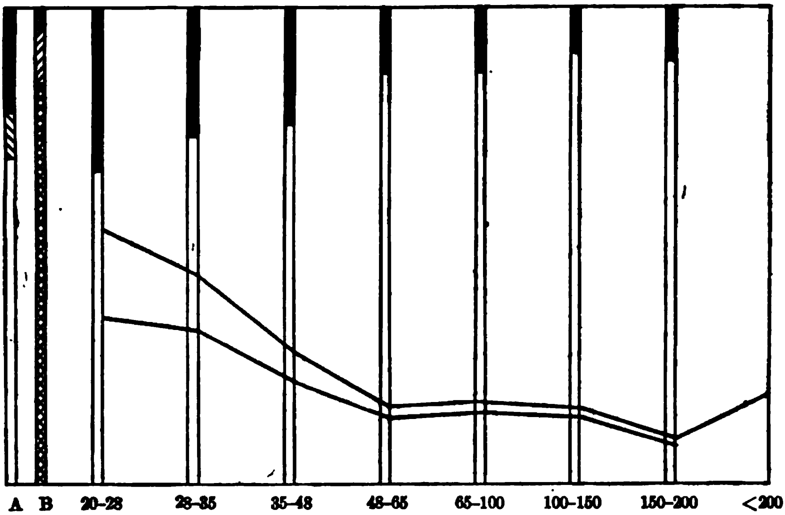

FIGURE 9.—Results of tests of sample 7.

SAMPLE 9. FRANK WHITE MINE.

Sample 9 was taken from the middle 6 feet of the Big seam, where it is cut by the incline at the top of the ridge.

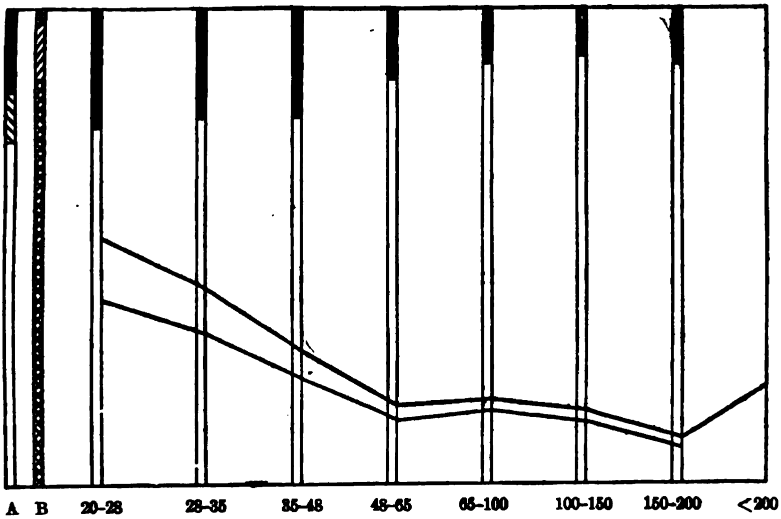

FIGURE 10.—Results of tests of sample 8.

SAMPLE 10. FRANK WHITE MINE.

Sample 10 was taken from the lowest 6 feet of the Big seam, where it is cut by the incline at the top of the ridge.

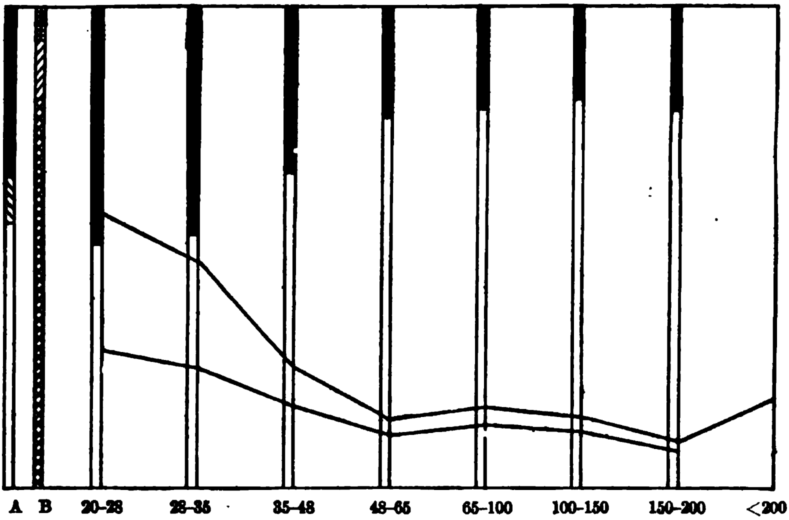

FIGURE 11.—Results of tests of sample 9.

No analyses were made of the products of this separation; but, as the ore was much finer grained than that from the overlying part of the bed, another portion of the sample was crushed to pass 35 mesh, and subjected to separation tests. The results are given on page 22.

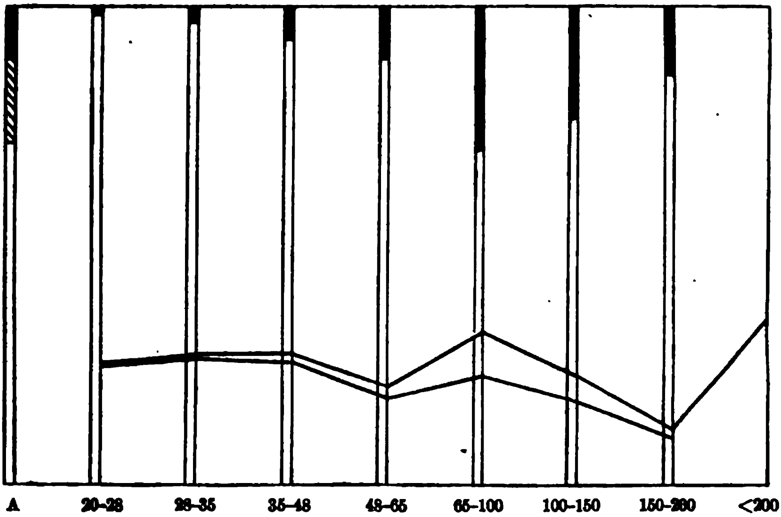

FIGURE 12.—Results of tests of sample 10.

As no analyses were made of the 20-mesh separation products, the composition of the products can not be compared. In the 35-mesh test the percentage of concentrates was reduced 8 per cent and percentage of tailings increased 6.4 per cent. Probably the concentrate was a little higher in grade and the slimes, on account of silica being crushed, a little lower. The screen-analysis curves show the same characteristics.

COMMENTS ON TESTS OF ORE FROM FRANK WHITE MINE.

In the separation tests the upper 6 feet of soft ore, averaging, in the two samples, 44.6 per cent Fe and 25.3 per cent SiO_2, gave a concentrate averaging 53 per cent Fe and 12.9 per cent SiO_2, with a ratio of concentration of 1.42; adding the slimes to the concentrates gives a product averaging 53 per cent Fe and 12.9 per cent SiO_2, with a concentration ratio of 1.25 and an increase in iron recovery from 84.5 to 96.2 per cent. Adding the slimes to the concentrates materially increases the iron recovery without lowering the grade of the concentrate. The iron content of the crude ore is about 6 per cent below the average for the soft ore of the district and about 7 per cent higher than the average in silica, and the ore can be utilized only when the iron trade is active. The tests produced from it a product equaling in grade the best ores obtainable in the district.

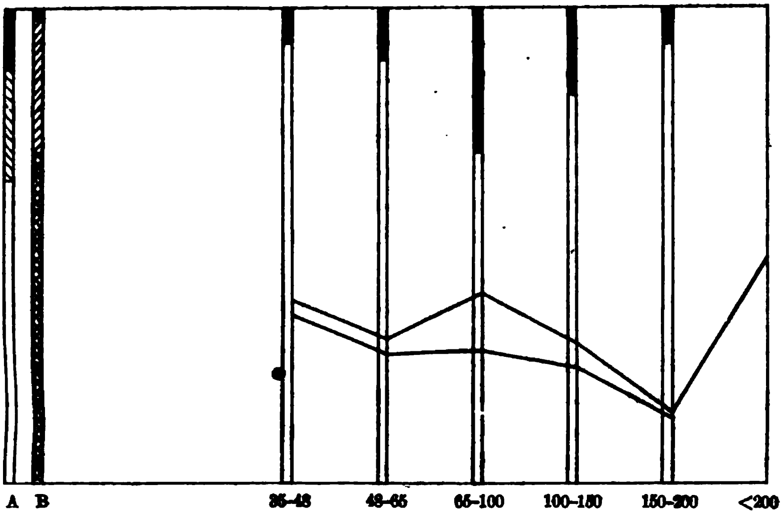

FIGURE 18.—Results of tests of sample 10 under 35 mesh.

The middle 6 feet of the Big seam is too low grade to be workable. The concentrates are poorer than the average soft ore of the district, and hence concentration by the method described would not be feasible for this part of the bed.

The composition of the lowest 6 feet of the bed is almost identical with that of the upper 6 feet, but the concentrates were no better than those from the middle 6 feet. The slimes from the lowest 6 feet are of much higher grade, however, and adding them to the concentrate gives a product slightly better than the average soft ore. The results are much poorer than those obtained with the upper 6 feet, and indicate that concentration of such ore is not practicable with the method used. The much finer grain of this ore, which prevents the silica from being separated to the same degree, accounts for the difference, although the ore was crushed to pass 35 mesh as against 20 mesh for ore from the upper 6 feet.

Averaging the analyses gives 42.2 per cent Fe and 30.5 per cent SiO_2 for the total thickness of the seam. Concentrates from it would carry 50.9 per cent Fe and 17.2 per cent SiO_2, with a ratio of concentration of 1.60 and an iron recovery of 78.2 per cent. Adding the slimes to the concentrate gives a product averaging 51.1 per cent Fe and 16.8 per cent SiO_2, with a ratio of concentration of 1.31 and an iron recovery of 95.5 per cent. Hence it seems that concentration of the entire bed, with an iron recovery of 95.5 per cent, would yield a product considerably better than the average of the soft ore of the district. Concentration would, therefore, make the entire seam workable.

ORE FROM THE LOWER BENCH, BIG SEAM.

There were tested 23 samples of ore from the lower bench of the Big seam from mines between Bessemer and Graces Gap and one from the Ruffner mine, about 10 miles northeast of Graces Gap. The 23 samples were obtained from that part of the district in which mining is most active, and, consequently, beneficiation of the siliceous ores is of immediate importance. Hence these ores have received more attention in this investigation than other ores and other parts of the district.

SAMPLE 11. RUFFNER MINE.

Sample 11 represents the upper 8 feet of the lower unworkable part (11 feet thick) and was taken in 2 left heading, 30 feet from the slope. It has the same fine grain as ore from the upper part of the seam, this feature being clearly shown by the screen-analysis curves (figs. 14 and 15).

Because of the ore being fine grained, the separation products were not analyzed, but another part of the sample was crushed to pass 35 mesh and tested, with the results given on page 22.

As the 20-mesh products were not analyzed, their composition can not be compared. The 35-mesh size yielded 8.5 per cent less concentrates, 5.6 per cent more slimes, and 2.9 per cent more tailings. This doubtless means that the concentrates of the 35-mesh size are appreciably better but the slimes are poorer than those of the 20-mesh size. The crude ore is of slightly lower grade than the two tested samples of crude ore from the upper part of the seam, yet the concentrate obtained runs 2 per cent higher in iron and nearly 1 per cent lower in silica than the average of that obtained from the other two samples; and the composition of concentrates plus slimes runs 2.2

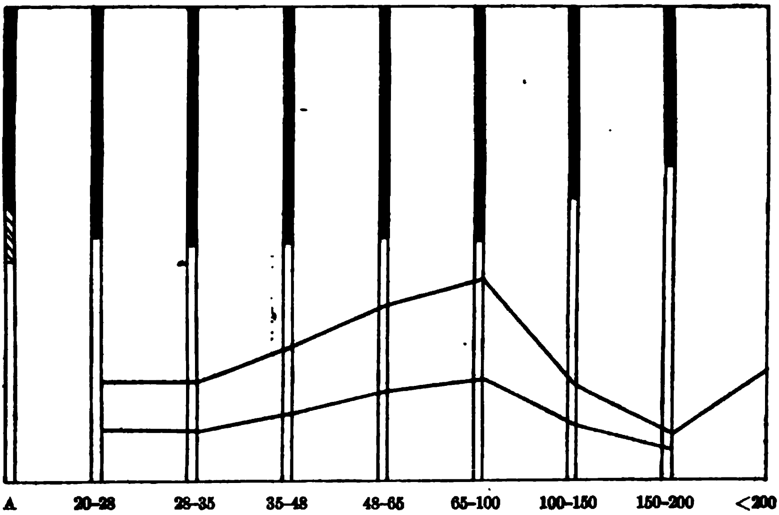

FIGURE 14.—Results of tests of sample 11.

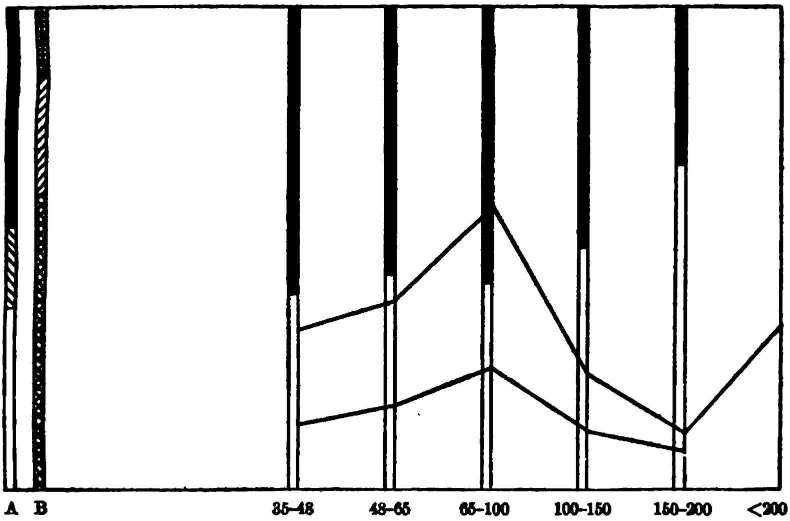

FIGURE 15.—Results of tests of sample 11 under 35 mesh.

per cent higher in iron and 1.3 per cent lower in silica than for the upper ores. As in the tests of the upper ore, however, the grade of the products is too low to make concentration attractive. The results would indicate that, if the Ruffner ore is to be concentrated, the entire bed will yield as good results as the upper 7 feet worked in the past.

SAMPLE 12. SPAULDING MINE.

In the Spaulding mine the Big seam is about 22 feet thick. No parting separates the seam into an upper and a lower bench, the upper more calcareous and less siliceous ore grading into the lower less calcareous and more siliceous ore. When the mine was active only the upper 11 feet of the seam was mined. Sample 12 represented the lower 11 feet of the seam and was taken in 1 left heading, cross left (a cross left is a branch to the left from a left main heading, and hence cuts well into the lower bench), 700 feet from the slope.

The concentrates and tailings from each mesh size were analyzed, and from the analyses the composition of the concentrates and tailings, as given on page 23, was calculated. The composition of the crude ore of each mesh size and the percentages of iron that went into the concentrates and tailings were calculated. The results are shown in the table below:

Composition of concentrates and tailings by mesh sizes.

Size of mesh.	Concentrates.		Tailings.		Crude.		Total iron in—	
	Fe.	SiO_2.	Fe.	SiO_2.	Fe.	SiO_2.	Concentrates.	Tailings.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
20 to 28	42.2	15.6	8.6	75.5	29.4	38.5	88.8	11.2
28 to 35	44.1	12.7	8.6	73.1	32.2	33.0	91.0	9.0
35 to 48	45.0	10.8	8.9	64.5	36.1	24.0	93.9	6.1
48 to 65	47.0	9.2	9.3	54.2	38.5	19.3	94.6	5.4
65 to 100	48.6	8.9	8.5	54.0	39.9	18.7	95.4	4.6
100 to 150	53.1	7.5	8.9	48.7	43.5	16.4	95.6	4.4
150 to 200	52.4	9.6	14.9	42.4	45.1	16.0	93.5	6.5
200					46.1	14.0		

The 20 to 28 and 28 to 35 mesh sizes are of lower grade than the original crude ore, whereas the finer sizes are all of higher grade, the quality improving with the fineness. If the 20 to 28 mesh size were rejected, the concentrate obtained from the remainder would average 46.7 per cent Fe and 10.6 per cent SiO_2, with a ratio of concentration of 2.07 and an iron recovery of 59.9 per cent. This product would run 1.2 per cent higher in iron and 1.3 per cent lower in silica, but the decrease in the iron recovery would be 23.2 per cent. Hence rejection of that size would not be warranted.

SAMPLE 13. SPAULDING MINE.

Sample 13 represents the upper 6 feet of the lower bench in 3 left heading, at the breast.

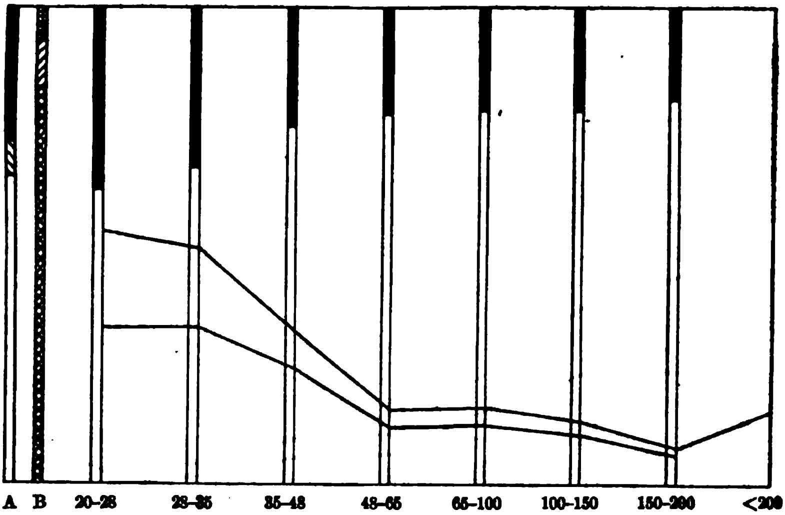

FIGURE 16.—Results of tests of sample 12.

SAMPLE 14. SPAULDING MINE.

Sample 14 was taken from the upper 4 feet of the lower bench of the Big seam in 5 left heading, 1,600 feet from the slope.

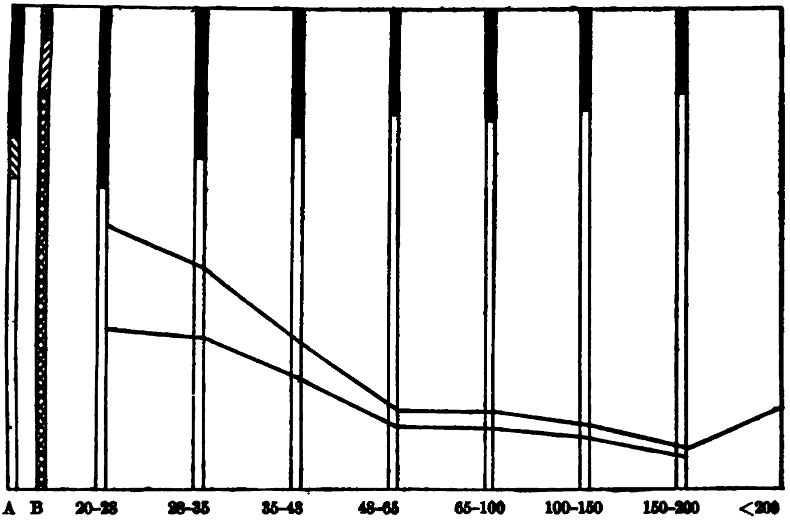

FIGURE 17.—Results of tests of sample 13.

Another part of the sample was crushed to pass 35 mesh and subjected to separation tests, with the results given on page 24. Only the concentrates were analyzed, they contained 45.3 per cent Fe and 13.0 per cent SiO_2, the ratio of concentration being 1.90 and the iron recovery 67.7 per cent. Compared with the 20-mesh test these concentrates have only 0.2 per cent more iron, and only 0.5 per cent less

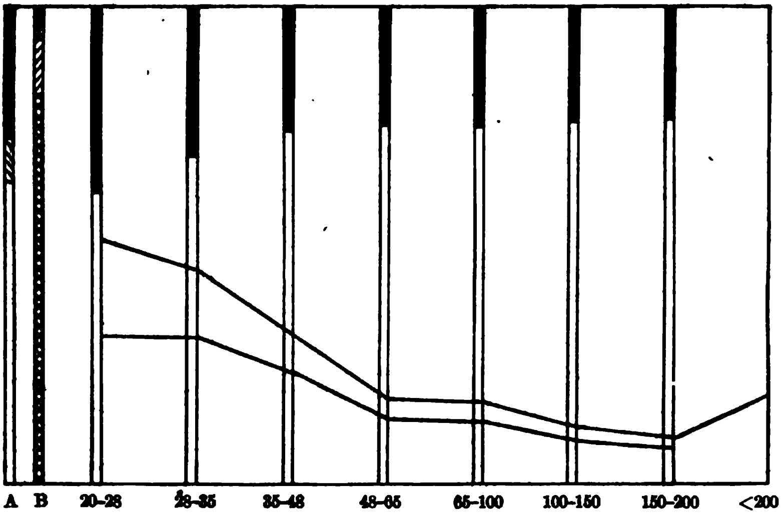

FIGURE 18.—Results of tests of sample 14.

silica, although the iron recovery is 13.7 per cent less. Such results would not justify the finer grinding. The amount of tailing is nearly the same for both 20 and 35 mesh ore, so that the lower percentage of concentrates is due almost entirely to the higher percentage of slimes.

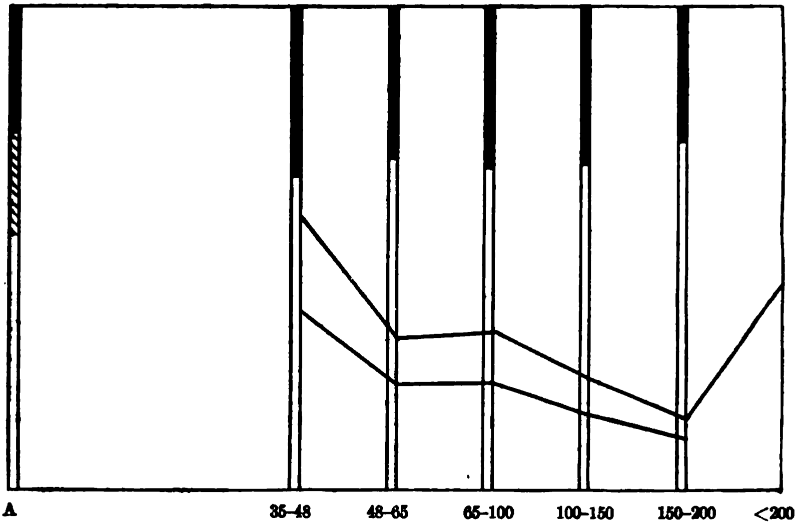

FIGURE 19.—Results of tests of sample 14 under 35 mesh.

SAMPLE 15. SPAULDING MINE.

Sample 15 was taken from the upper 3 feet of the lower bench of the Big seam in 6 left heading, at the breast of the drift.

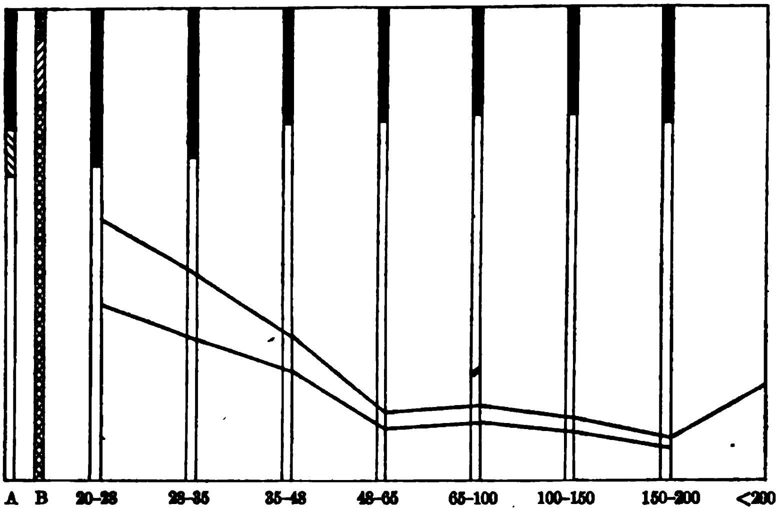

FIGURE 20.—Results of tests of sample 15.

SAMPLE 16. SPAULDING MINE.

Sample 16 was taken from the 3 feet of ore immediately below the workable ore, in 10 left heading, at the breast.

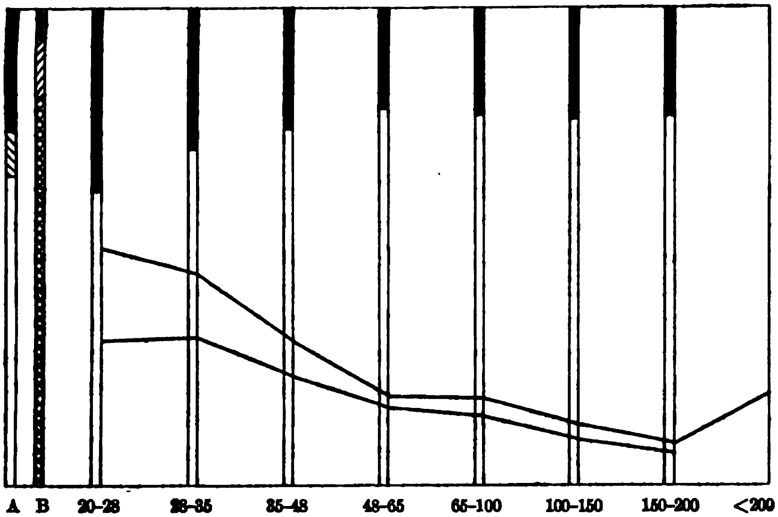

FIGURE 21.—Results of tests of sample 16.

SAMPLE 17. SPAULDING MINE.

Sample 17 was taken from the upper 8 feet of the lower bench of the Big seam in 6 right heading, 600 feet from the slope.

COMMENTS ON TESTS OF LOWER BENCH BIG SEAM ORE FROM THE SPAULDING MINE.

The results of the tests of the preceding six samples of ore are strikingly uniform. The average of the results obtained from the six samples is given in Table 1 on page 38. They deviate only a little from the results obtained with the individual samples, hence the Spaulding lower bench ore is presumably of uniform character throughout, and the results obtained may be regarded with more than usual confidence as representing what may be expected of this ore under large-scale treatment. The slimes are so little lower in grade than the concentrates that in order to increase the iron recovery 11.4 per cent the mixture of concentrates plus slimes can be considered as the final product. Then concentration increases the iron 9.5 per cent, reduces the silica 13.7 per cent, and, judged by the single determination, leaves the percentage of lime practically unchanged, the net result being a decided increase in quality, with a concentration of about 4 tons into 3 and an iron recovery of 93.3 per cent. Such a product is better than the average washed brown ore of the district. Burchard[a] gives 23 analyses of washed brown

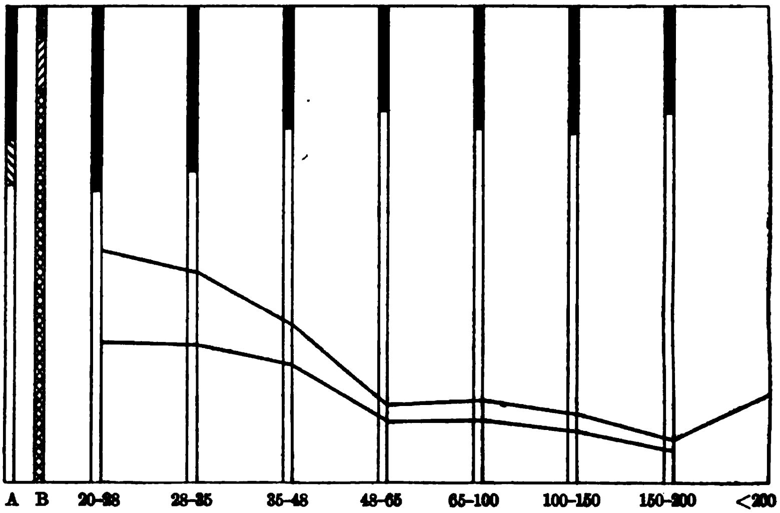

FIGURE 22.—Results of tests of sample 17.

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[a] Burchard, E. F., and Butts, Charles, Iron ores, fuels, and fluxes of the Birmingham district, Ala.: U. S. Geol. Survey Bull. 400, 1910, p. 169.

ore from the Woodstock and Champion areas, which average 45 per cent Fe, 14.6 per cent SiO_2, 4 per cent Al_2O_3, and 0.74 per cent Mn. The Spaulding concentrate runs 0.6 per cent higher in iron and 1.5 per cent lower in silica and its lime content more than counterbalances the manganese content of the brown ore. According to the results, concentration of the lower bench Spaulding ore should produce a product that can easily compete with the brown ores of the district.

SAMPLE 18. ISHKOODA MINE.

The upper bench in the Ishkooda mine is about 7 feet thick. A mere parting along the bedding plane with only here and there a thin layer of shale separates it from the lower bench. In working this bench about 3 feet and sometimes as much as 5 feet of the lower bench ore is also taken. From 7 to 10 feet of ore is left beneath and sample 18 was taken from it in 18 left heading, 200 feet from the slope.

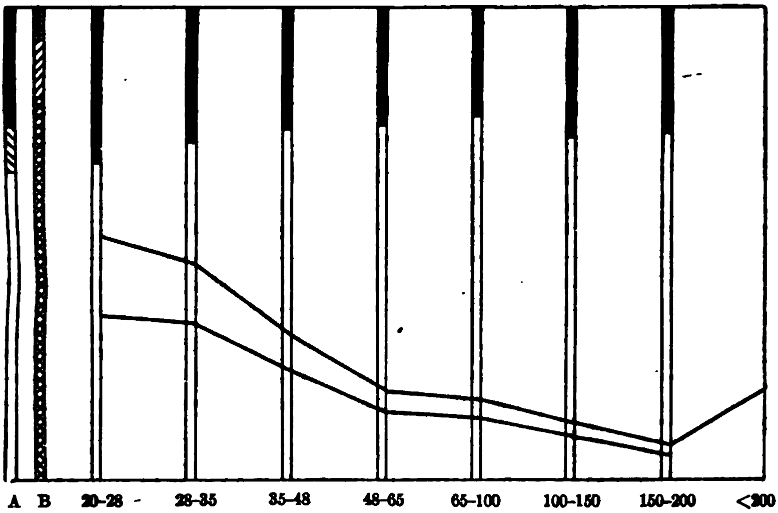

FIGURE 23.—Results of tests of sample 18.

SAMPLE 19. ISHKOODA MINE.

Sample 19 was taken from the upper 4 feet of the lower bench of the Big seam at the same place as sample 18 and represents lower bench ore that is mined with upper bench ore.

SAMPLE 20. ISHKOODA MINE.

Sample 20 was taken in 15 right heading, 500 feet from the slope, and represents the lower bench 8 to 10 feet below the parting.

The concentrates and tailings of each mesh size were analyzed separately and the composition of the concentrates and tailings for the entire sample, as given in Table 1 on page 26, was calculated from these. The table below shows these analyses and the calculated composition of the crude of each mesh size.

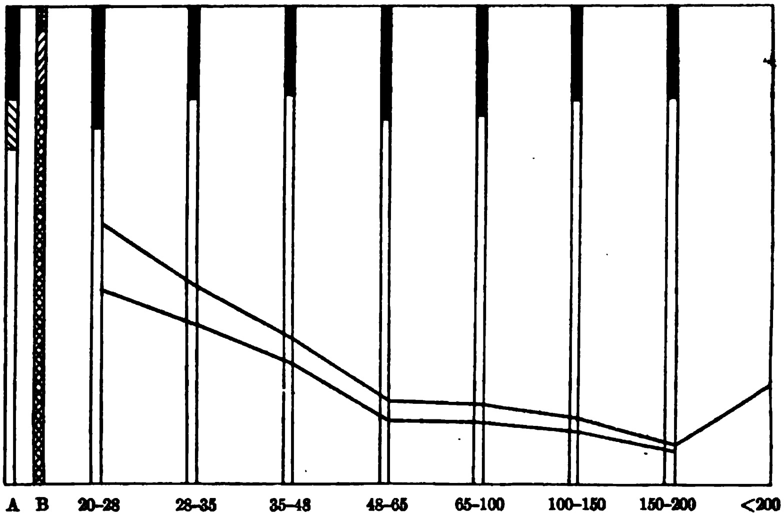

FIGURE 24.—Results of tests of sample 19.

Composition of concentrates and tailing by mesh sizes.

Size of mesh.	Concentrates.		Tailings.		Crude.		Total iron in—	
	Fe.	SiO_2.	Fe.	SiO_2.	Fe.	SiO_2.	Concentrates.	Tailings.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
20 to 28	43.5	19.3	8.7	68.5	28.8	40.1	87.3	12.7
28 to 35	37.2	21.7	9.1	63.3	29.6	33.1	91.6	8.4
35 to 48	42.2	12.4	8.7	51.8	31.8	24.7	91.5	8.5
48 to 65	43.3	10.4	8.1	41.6	32.4	20.1	92.3	7.7
65 to 100	46.6	9.2	8.0	36.8	33.9	18.3	92.2	7.8
100 to 150	47.9	9.7	8.9	33.3	36.3	16.7	92.7	7.3
150 to 200	50.7	8.6	13.1	29.3	43.3	12.7	94.0	6.0
200	....	....	....	....	39.8	12.4	....	....

The 20 to 28 and 28 to 35 mesh sizes are of lower grade than the original ore, whereas all smaller sizes of the crude are of higher grade, the quality increasing with the fineness, except that the 150 to 200 mesh size contains more iron than the slimes, and is higher in iron and lower in silica than the concentrates themselves. Hence, everything smaller than 150-mesh might have been added directly to the final product.

Another part of the same sample was crushed to pass 35-mesh and subjected to separation tests, with the results given on page 26. By analysis these concentrates contained 42.1 per cent Fe and 13.3 per cent SiO_2, the silica being nearly 3 per cent lower than in the concentrates of the 20-mesh test. Compared with that test the ratio of concentration is 2.10 instead of 1.66, and the iron recovery 61.9 as against 80 per cent. Finer crushing increased the proportion of tailings only a little more than 1 per cent, and the decrease of concentrates, 12.8 per cent, is offset by the larger proportion of slimes. Adding the slimes to the concentrates yields a final product that would probably differ little from the corresponding product of the 20-mesh test.

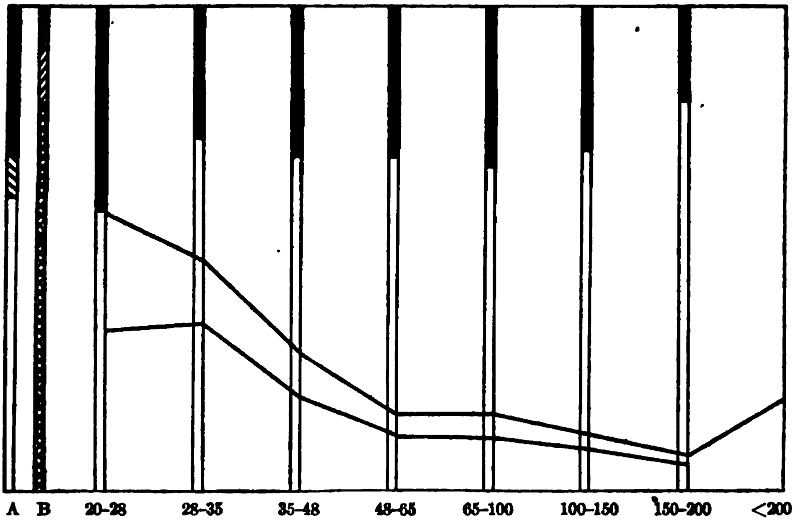

Figure 25.—Results of tests of sample 20.

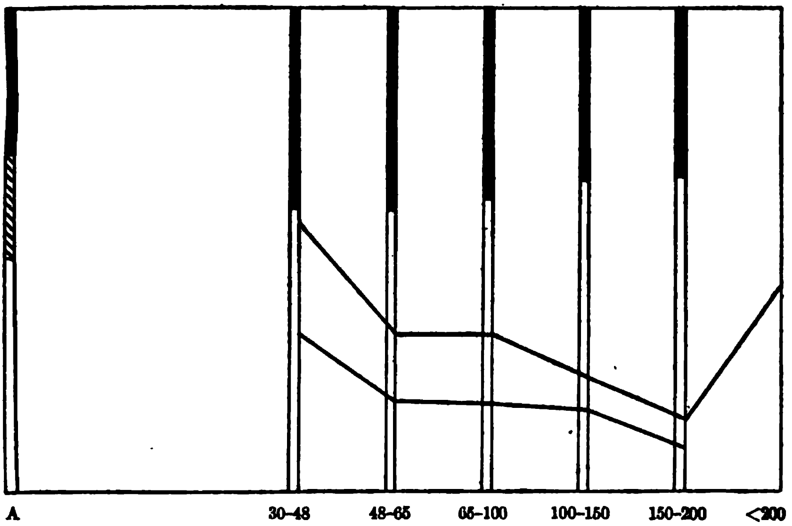

Figure 26.—Results of tests of sample 20 under 35 mesh.

SAMPLE 21. ISHKOODA MINE.

Sample 21 was taken in 6 right heading, near the manway. Here the parting is a 1 to 2 inch layer of shale, and the sample was taken from the $3\frac{1}{2}$ feet of ore just under it.

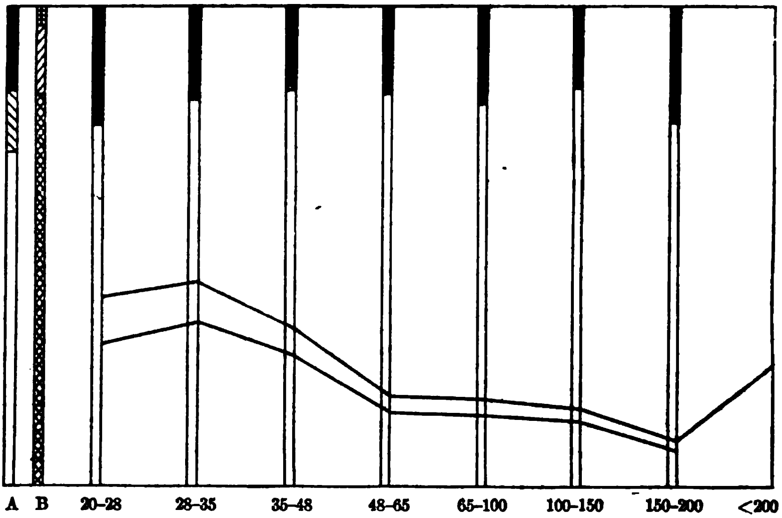

FIGURE 27.—Results of tests of sample 21.

SAMPLE 22. ISHKOODA MINE.

Sample 22 was taken from the upper 7 feet of the lower bench in 10 right heading, 200 feet from the slope. Here the parting is merely a break in the bedding, no shale being present.

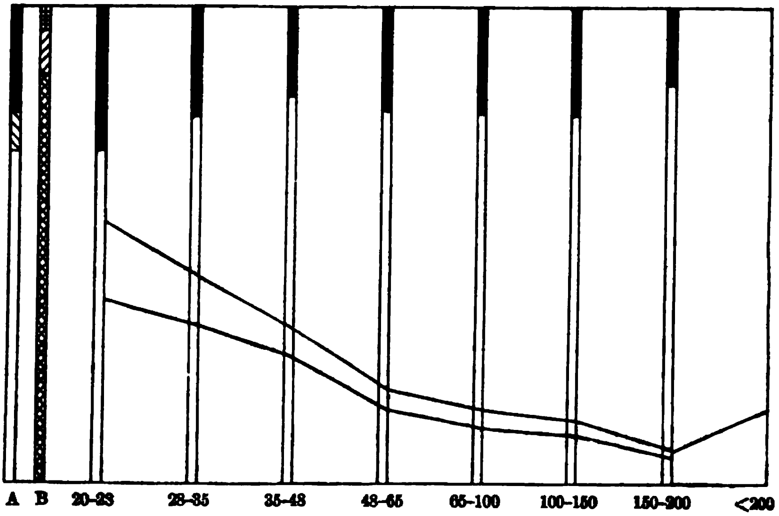

FIGURE 28.—Results of tests of sample 22.

COMMENTS ON TESTS OF ORE FROM LOWER BENCH IN ISHKOODA MINE.

Compared with the Spaulding ore, the five samples show considerable variation, largely because samples 20 and 21 represent only the less siliceous part of the lower bench ore that is taken out with the upper bench in mining. In consequence the averages of the five samples of Ishkooda ore, on page 37, are somewhat better than the probable average of the whole bench.

The composition of the slimes is so nearly like that of the concentrates that only a final product of slimes plus concentrates need be considered, this product being of the same grade and the iron recovery being higher. This product is 8.5 per cent higher in iron and 13.3 per cent lower in silica than the original ore and has about the same lime content according to the one determination. Compared with the average of the brown ores of the district (p. 76), this product runs 0.6 per cent higher in iron, 2.3 per cent lower in silica, and has a lime content of nearly 8 per cent to offset the manganese content of the brown ores. Judged by its composition, it can readily compete with them. As samples 19 and 21 represent only the best part of the lower bench, the results from samples 18, 20, and 22 were also averaged separately. The composition of the concentrates plus slimes averaged 44.0 per cent Fe and 13.6 per cent SiO_2, or 1 per cent less in iron and silica than the brown ores, and hence compares favorably with them. This result is obtained with a concentration of 4 tons into 3 and an iron recovery of 93.2 per cent.

SAMPLES FROM SONGO MINE.

In this mine the upper bench of the Big seam is 7½ to 9 feet thick and is separated from the lower bench by a 6 to 9 inch shale parting. The lower bench is about 12 feet thick, its upper 9 feet being relatively free from shale. At only one point, in 14 right heading, was the lowest 3 feet cut into, and there it consisted of intercalated shale and ore. Down to heading 23, the upper 9 feet of the lower bench has been cut into in the headings and the ore used by the Woodward Iron Co., which works this mine, in its own furnaces; but the quality is such that the ore can not be sold in the open market. Nearly all the ore mined is from above the shale parting.

SAMPLE 23. SONGO MINE.

Sample 23 was taken in 14 right heading, about 600 feet from the slope; at this point the entire lower bench is exposed, but on account of the lower 3 feet being shaly only the upper 9 feet were sampled.

SAMPLE 24. SONGO MINE.

Sample 24 represents the upper 6 feet of the lower bench in 12 left heading, 250 feet from the slope.

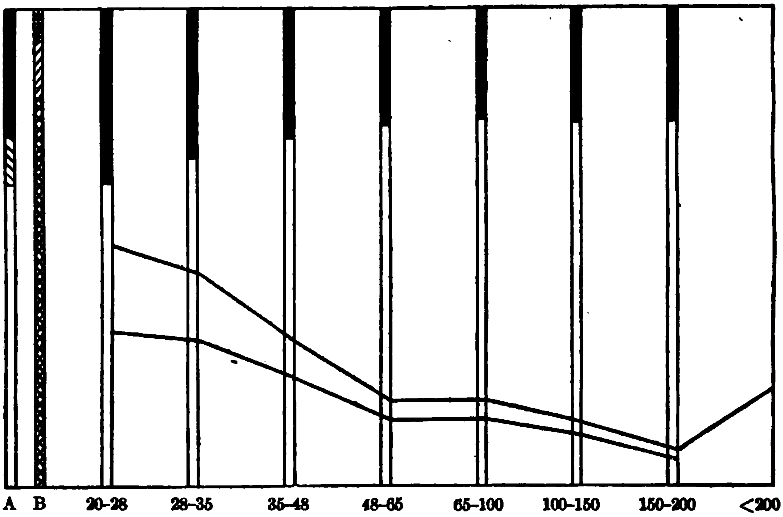

FIGURE 29.—Results of tests of sample 23.

SAMPLE 25. SONGO MINE.

Sample 25 was taken from the upper 9 feet of the lower bench in 15 left heading, 300 feet from the slope.

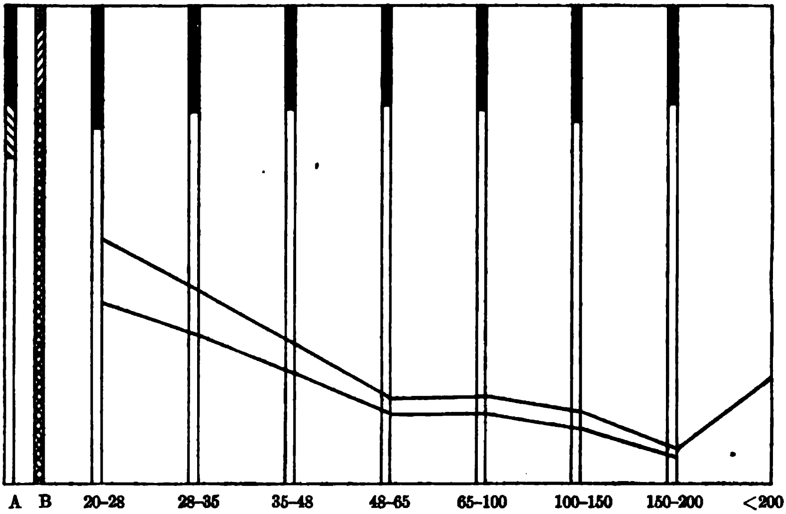

FIGURE 30.—Results of tests of sample 24.

SAMPLE 26. SONGO MINE.

Sample 26 was taken from the upper 9 feet of the lower bench of the Big seam in 18 right heading, 700 feet from the slope.

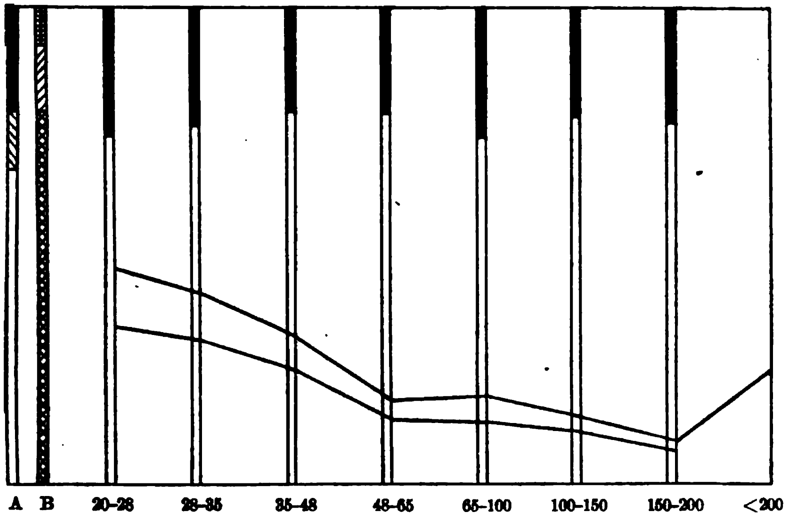

FIGURE 31.—Results of tests of sample 25.

SAMPLE 27. SONGO MINE.

Sample 27 was taken from the upper 9 feet of the lower bench in 19 left heading, 300 feet from the slope.

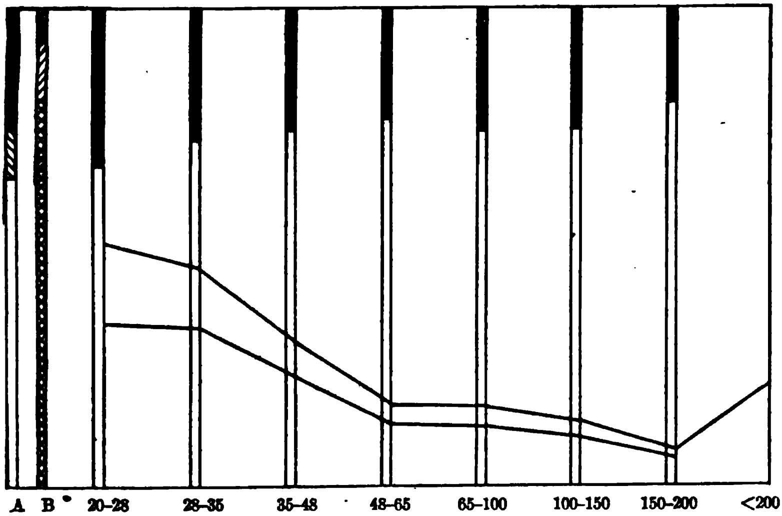

FIGURE 32.—Results of tests of sample 26.

Another portion of the sample was crushed to pass 35 mesh and subjected to separation tests, with the results given on page 29

The concentrates from this separation analyzed 45.7 per cent Fe and 11.0 per cent SiO_2, with a ratio of concentration of 1.79 and an iron recovery of 68.5 per cent. Finer crushing consequently produced a concentrate 2.2 per cent higher in iron and lower in silica, but reduced the iron recovery from 83.2 to 68.5 per cent. The proportion of tailings was nearly the same in the two tests, and the slimes increased at the expense of the concentrates.

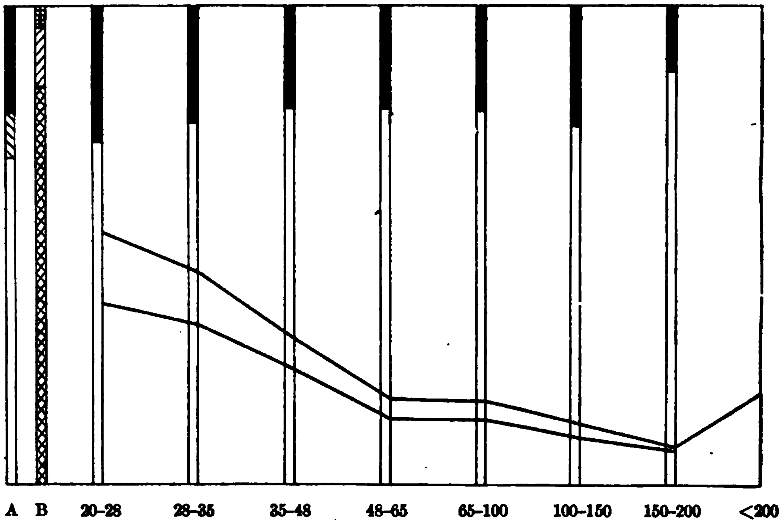

FIGURE 33.—Results of tests of sample 27.

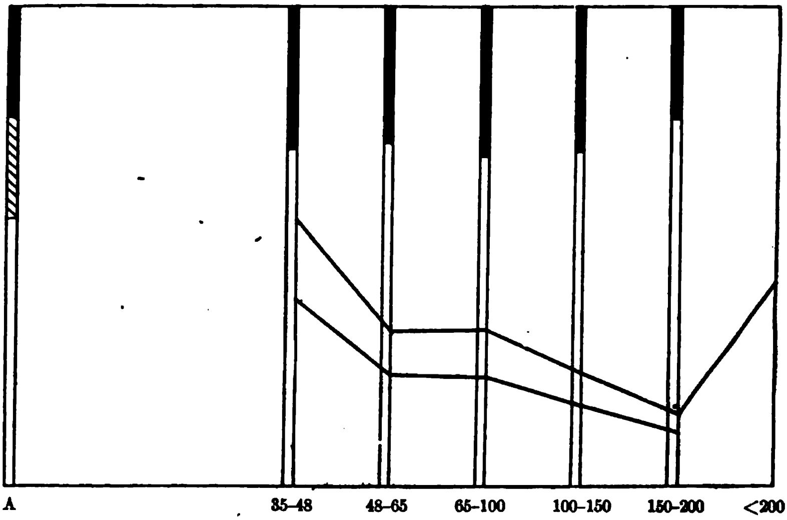

FIGURE 34.—Results of tests of sample 27 under 35 mesh.

SAMPLE 28. SONGO MINE.

Sample 28 was taken from the upper 8 feet of the lower bench of the Big seam in 23 left heading, 150 feet from the slope.

The concentrates and tailings of each mesh size were analyzed separately and the composition of the entire concentrates and tailings calculated from these analyses, which are given in the table below, with the calculated composition of the crude ore of each size.

Composition of concentrates and tailings by mesh sizes.

Size of mesh.	Concentrates.		Tailings.		Crude.		Total iron in—	
	Fe.	SiO$_2$.	Fe.	SiO$_2$.	Fe.	SiO$_2$.	Concentrates.	Tailings.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
20 to 28	41.5	15.4	8.3	65.3	31.9	29.9	92.4	7.6
28 to 35	43.8	12.4	8.2	62.6	35.2	24.4	94.4	5.6
35 to 48	44.6	11.1	8.1	54.7	37.3	19.9	95.6	4.4
48 to 65	54.8	9.9	8.3	46.8	38.3	17.3	95.7	4.3
65 to 100	49.5	8.8	7.9	43.1	38.7	16.2	95.6	4.4
100 to 150	53.0	7.7	6.9	42.9	43.0	15.2	96.6	3.4
150 to 200	54.4	8.1	7.1	41.8	45.3	14.6	97.0	3.0
200	....	....	....	....	47.4	11.7	....	....

Mesh sizes larger than 48 mesh are of lower grade and those smaller than 48 mesh are of higher grade than the original ore, each size being of higher grade than the preceding. Of the crude ore sizes, only the slimes are better than the concentrates, but the crude 150 to 200 mesh ore runs better than the concentrate of the 20 to 28 mesh size.

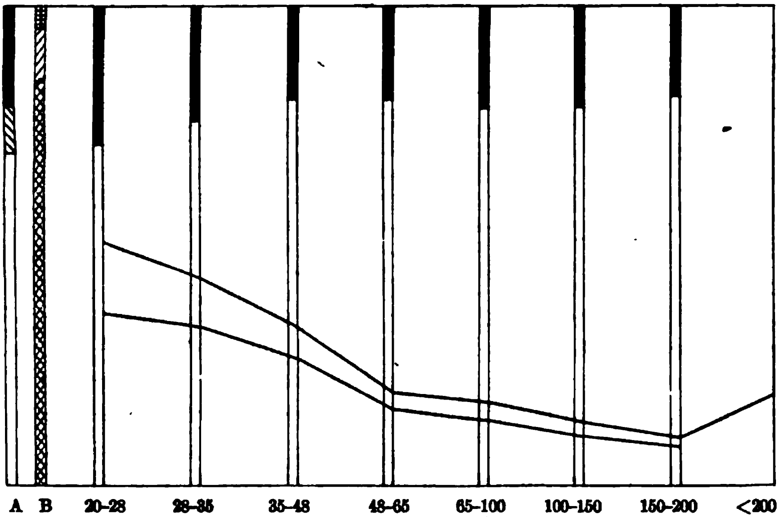

FIGURE 35.—Results of tests of sample 28.

COMMENTS OF TESTS OF ORE FROM SONGO MINE.

The six samples from the lower bench show a uniformity of composition almost equal to that of the Spaulding ore. Hence the bed at the points of sampling is of uniform grade chemically, and also, as shown by the separation tests, physically. Such uniformity of character indicates that in actual practice there would probably be little deviation from these results. The average of the results obtained with the six samples is given in the table on page 38.

An average of several analyses supplied by Mr. A. H. Woodward of lower bench Songo ore is 39.5 per cent Fe, 20 per cent insoluble, and 10.4 per cent CaO. These figures indicate that the samples tested are representative of the ore. The slimes practically average the same as the concentrates, therefore, in view of the increased iron recovery, the final product may be considered as concentrates plus slimes. Such a product on the basis of 4 tons into 3, will give an iron recovery of 94.3 per cent, and be 6.6 per cent higher in iron, 8.7 per cent lower in silica, and nearly as high in lime as the crude ore.

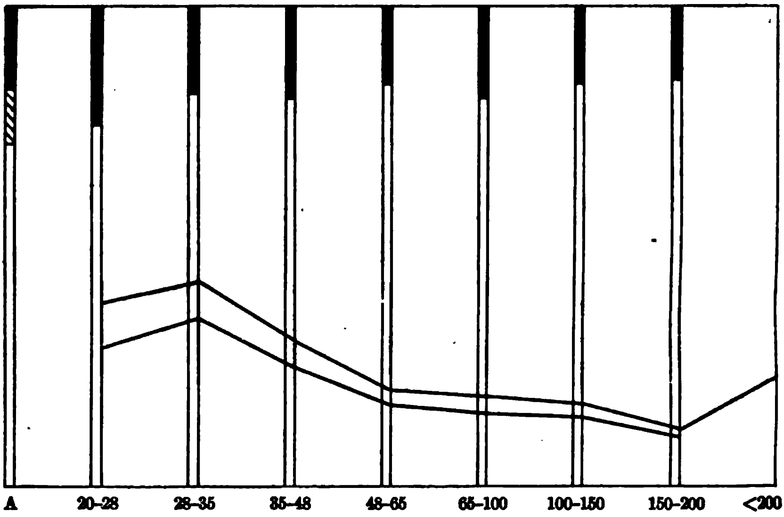

FIGURE 36.—Results of tests of sample 29.

Compared with the average of the brown ores of the district (p. 76), the product has the same iron content, 2 per cent less silica, and a high lime content to offset the manganese of the brown ores. On the basis of its chemical composition it can compete with those ores.

ORE FROM FOSSIL MINE.

SAMPLE 29. FOSSIL MINE.

In this mine the upper bench only of the Big seam is worked. Sample 29 represents the upper 4 feet of the lower bench, in 10 slope, 24 left heading, 100 feet from the slope.

Only the concentrates were analyzed, and they carried 48 per cent Fe and 12.1 per cent SiO_2. Another portion of the same sample was crushed to pass 35 mesh and subjected to separation tests, with the results given on page 30.

As the composition of the slimes is so much lower than that of the concentrates, adding the slimes to the concentrates would lower the grade of the final product. Yet as the iron recovery would be increased and the resulting product would still be slightly higher in iron and lower in silica than the average of the brown ores, the procedure would probably be found practicable. The 20 mesh concentrate is practically as good as the 35 mesh, and was obtained with a ratio of concentration of 1.40 as against 1.71, and an iron recovery of 86.2 per cent as against 71.1 per cent. There seems to be no advantage, therefore, in crushing this ore finer than 20 mesh. At least, 35 mesh is not fine enough to show an appreciable advantage.

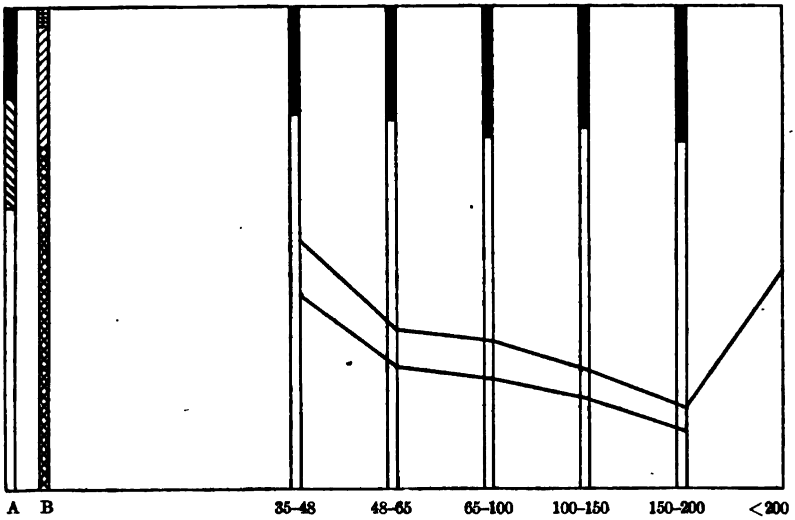

FIGURE 37.—Results of tests of sample 29 under 35 mesh.

SAMPLE 30. FOSSIL MINE.

Sample 30 was taken in 10 slope, 31 left heading, 125 feet from the slope. The lower bench here is a little more than 8 feet thick, and the sample represents the upper 8 feet of it.

SAMPLE 31. FOSSIL MINE.

Sample 31 was taken at 31 heading in the bottom of 10 slope. At that place the parting of the two benches comprises two ½-inch bands of shale, with a 4-inch layer of ore between. The lower bench is 9 feet thick, and the sample represents the entire thickness.

SAMPLE 32. FOSSIL MINE.

Sample 32 represents the entire thickness of the lower bench in 9½ slope, 23 left heading, 30 feet from the slope.

SAMPLE 33. FOSSIL MINE.

Sample 33 represents the whole thickness of the lower bench, on the north side of 9 slope between headings 47 and 49.

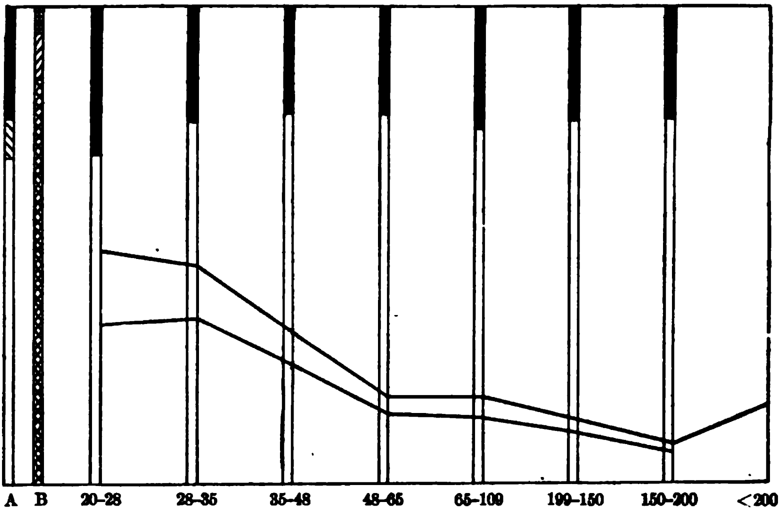

FIGURE 38.—Results of tests of sample 30.

The concentrates and tailings from the different mesh sizes were analyzed separately, and the composition of the concentrates and

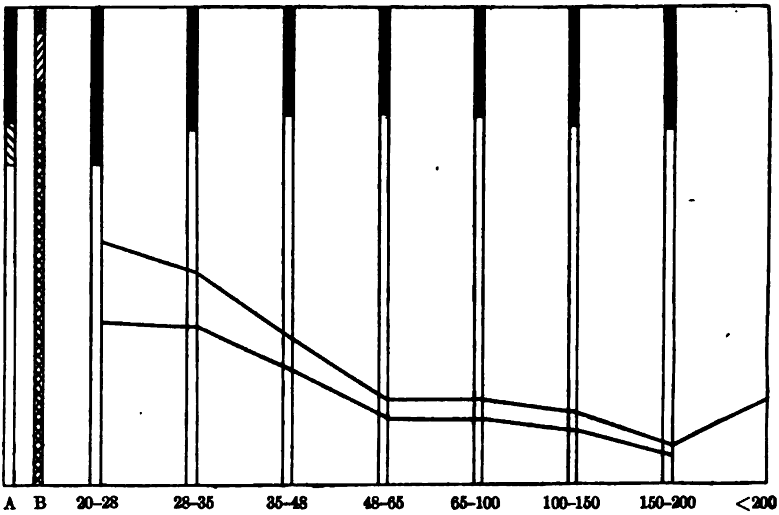

FIGURE 39.—Results of tests of sample 31.

tailings, as given on page 31, was calculated from these analyses. The calculated composition of the crude ore of each size and the percentage of iron that went into the concentrates and tailings of each are given in the accompanying table.

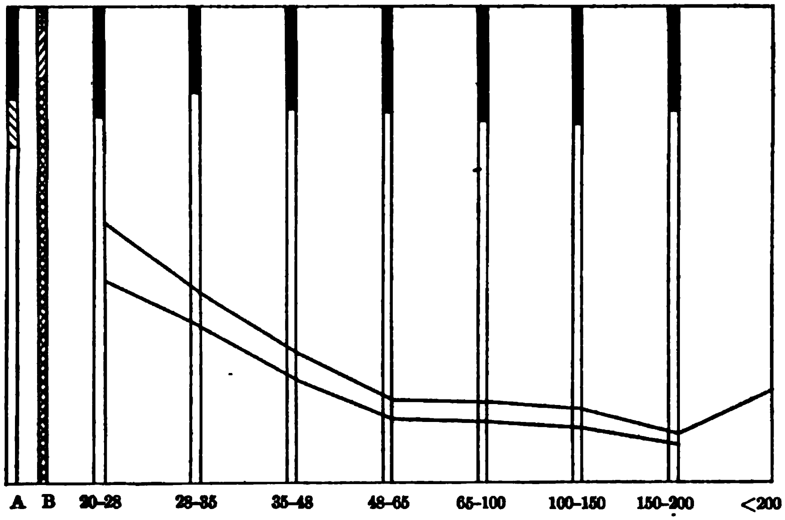

FIGURE 40.—Results of tests of sample 32.

Composition of concentrates and tailings by mesh sizes.

Size of mesh.	Concentrates.		Tailings.		Crude.		Total iron in—	
	Fe.	SiO_2.	Fe.	SiO_2.	Fe.	SiO_2.	Concentrates.	Tailings.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
20 to 28	42.4	15.6	8.2	70.9	32.1	32.3	92.3	7.7
28 to 35	44.4	12.7	8.6	63.3	36.7	23.8	94.9	5.1
35 to 48	44.3	11.5	8.5	53.5	37.4	19.7	95.6	4.4
48 to 65	45.9	9.9	7.9	41.9	38.6	16.0	96.0	4.0
65 to 100	48.3	8.9	8.0	37.1	39.9	14.8	95.8	4.2
100 to 150	50.6	8.6	10.2	30.9	42.4	13.2	95.1	4.9
150 to 200	54.9	7.5	10.4	32.9	44.7	13.3	94.7	5.3
200	....	....	....	....	45.5	11.3	....	....

In this sample the 35 to 48 mesh size has about the same grade as the crude ore, and the coarser sizes are poorer. The finer sizes improve in quality down to the fines. The slimes are the only size of the crude ore that runs better than the concentrates, but all the crude smaller than 100 mesh runs better than the concentrates of the 20 to 28 mesh size.

SAMPLE 34. FOSSIL MINE.

Sample 34 represents the entire lower bench in 8 slope between headings 48 and 50.

COMMENTS ON TESTS OF ORE FROM FOSSIL MINE.

As the results for sample 29 were obtained by crushing the ore to pass 35 mesh, and those for the rest of the samples by crushing to pass 20 mesh, they are omitted in considering the average results (p. 39). The crude ore has the same composition as the other five samples, and the better products doubtless result from finer crushing, although the concentrate of the 20-mesh test runs a little higher than the concentrates from the rest of the samples.

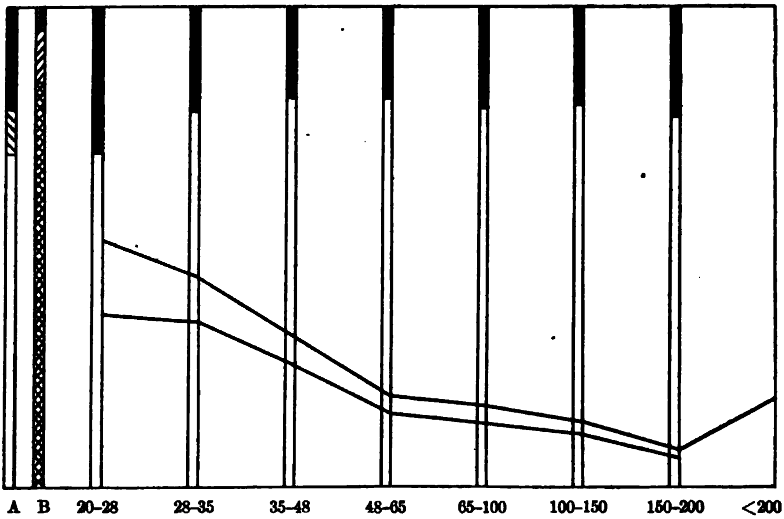

FIGURE 41.—Results of tests of sample 33.

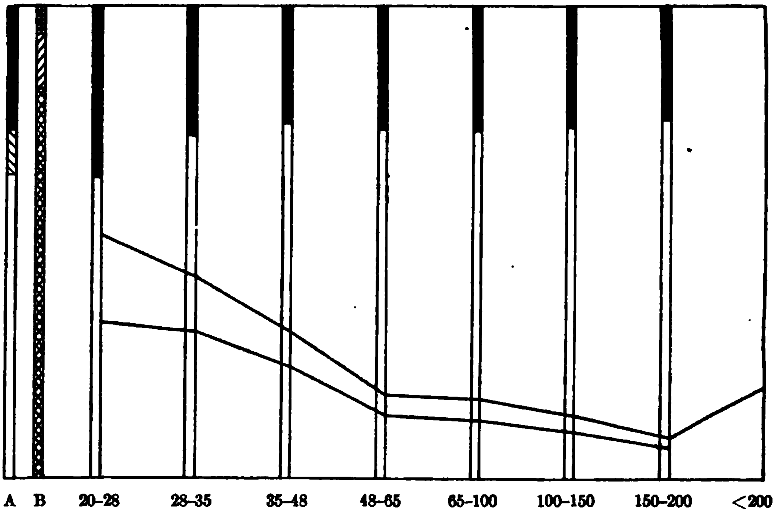

FIGURE 42.—Results of tests of sample 34.

An average of eight analyses of lower bench Fossil ore given by Burchard[a] is 37.3 per cent Fe, 20.2 per cent SiO_2, 3.3 per cent Al_2O_3, and 11.9 per cent CaO. This is very nearly the same as the average of samples 30 to 34, as given in Table 2 on page 39. The individual samples depart little from the average and show the uniformity of results that characterizes the separation tests. The ore is, therefore, shown to be quite uniform in chemical composition and physical character throughout the mine.

As the grade of the slimes is so little below that of the concentrates, it is advantageous to consider the final product as concentrates plus slimes. Then a concentration in the ratio of 4 tons into 3 yields an iron recovery of 94.8 per cent and a product 7.7 per cent higher in iron, 8.6 per cent lower in silica, and, on the basis of the one analysis, only 2 per cent lower in lime. Compared with the average of the washed brown ores of the district, such a concentrate would be 0.3 per cent richer in iron, 2.4 per cent lower in silica, and have an appreciable lime content to offset the value of the manganese in the brown ore. Hence, on the basis of its chemical composition it should be able to compete favorably with those ores.

SUMMARY OF COMMENTS ON TESTS OF LOWER BENCH BIG SEAM ORE.

On pages 50–71 are given the results of tests with 24 samples of ore from the lower bench of the Big seam. Sample 11 from the Ruffner mine represents very low grade ore and its wet concentration under present conditions does not seem justifiable.

The remaining 23 samples represent ore from different points in four important mines. Sample 29, from the Fossil mine, was crushed to 35 mesh, and the complete results are for that mesh size; as complete results for other samples were determined with the 20-mesh crushing, sample 29 is omitted from the discussion of the average results. Finer crushing of sample 29 had little effect, however, in improving the grade of the concentrate, as the 20-mesh concentrate contained only 0.1 per cent less iron and 0.4 per cent more silica than ore crushed to pass 35 mesh, and the iron recovery was 15 per cent higher. Hence there was no advantage in crushing this ore finer.

The average of the results obtained with the remaining 22 samples is given in the accompanying table. The column marked concentrates included the concentrates plus slimes.

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[a] Burchard, E. F., and Butts, Charles, Iron ores, fuels, and fluxes of the Birmingham district, Alabama, with chapters on the origin of the ores, by E. C. Eckel: U. S. Geol. Survey Bull. 400, 1910, pp. 83–84.

COMMENTS ON TESTS OF ORE FROM FOSSIL MINE.

As the results for sample 29 were obtained by crushing the ore to pass 35 mesh, and those for the rest of the samples by crushing to pass 20 mesh, they are omitted in considering the average results (p. 39). The crude ore has the same composition as the other five

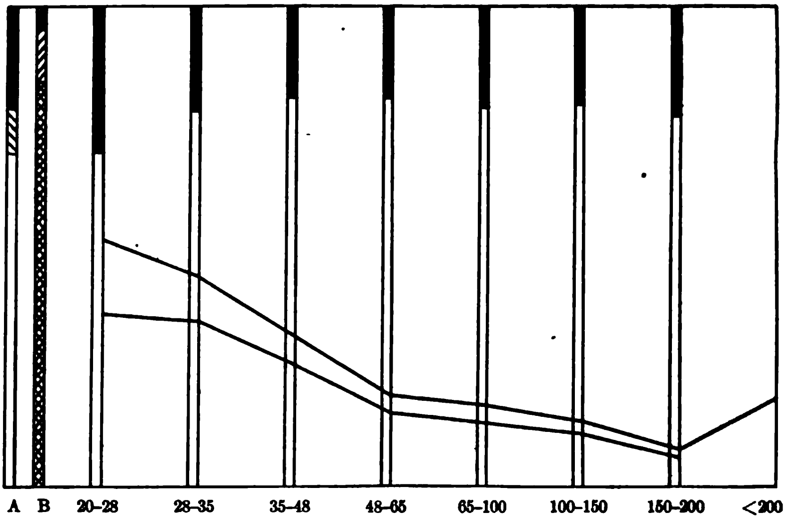

FIGURE 41.—Results of tests of sample 33.

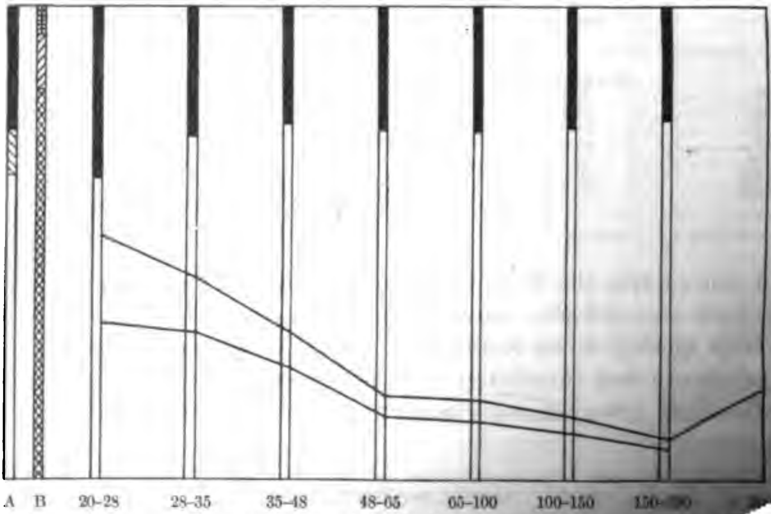

FIGURE 42.—Results of tests of sample 34.

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An average of eight analyses of lower bench [illegible] Burchard[a] is 37.3 per cent Fe, 20.2 per cent [illegible] and 11.9 per cent CaO. This is very nearly the same [illegible] of samples 30 to 34, as given in Table 2 on page [illegible] samples depart little from the average and show [illegible] results that characterizes the separation tests. [illegible] shown to be quite uniform in chemical [illegible] character throughout the mine.

As the grade of the slimes is so little below [illegible] trates, it is advantageous to consider the [illegible] plus slimes. Then a concentration in the [illegible] an iron recovery of 94.8 per cent and a product [illegible] iron, 8.6 per cent lower in silica, and, on the [illegible] only 2 per cent lower in lime. Compared [illegible] washed brown ores of the district, such a [illegible] per cent richer in iron, 2.4 per cent lower [illegible] appreciable lime content to offset the value of [illegible] brown ore. Hence, on the basis of its [illegible] be able to compete favorably with those ores.

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The average [illegible] with the remaining 23 samples is given i[illegible] The column marked trates i[illegible] slimes.

[a] [illegible]

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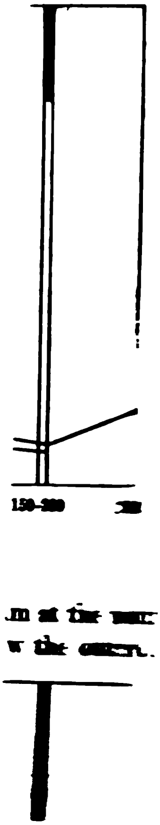

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Average results of tests.

Mine.	Crude ore.			Concentrates.			Recovery of iron.	Ratio of concentration.
	Fe.	SiO_2.	CaO.	Fe.	SiO_2.	CaO.		
	P.ct.	*P.ct.*	*P.ct.*	*P.ct.*	*P.ct.*	*P.ct.*	*P.ct.*	
Spaulding [a]	36.1	26.8	7.5	45.6	13.1	7.6	93.3	1.37
Ishkooda [b]	37.1	25.6	8.0	45.6	12.3	7.7	94.1	1.31
Songo [a]	38.4	21.2	10.5	45.0	12.5	9.5	94.3	1.30
Fossil [b]	37.6	20.8	9.6	45.3	12.2	7.6	94.8	1.30
Average of the four groups	37.3	23.6	8.9	45.4	12.5	8.1	94.1	1.32
Average composition of washed brown ores	....	....	....	45.0	14.6	....	....	....

[a] Average of six samples. [b] Average of five samples.

As the results from the 22 samples are decidedly uniform, the calculated average in the table may be safely taken as representing what one may expect from beneficiating these ores under the conditions of the experiments. The final product obtained by concentrating 4 tons of crude ore into 3, with an iron recovery of 94.1 per cent, runs 0.4 per cent higher in iron, and 2.1 per cent lower in silica than the brown ores, and has a considerable lime content to enhance its value.

ORE FROM THE IRONDALE SEAM.

The Irondale seam is of most value along the northeastern part of Red Mountain, from Pilot Knob southwest to a point about opposite Birmingham, a distance of about 10 miles. Farther southwest the bed becomes poorer, showing interstratified bands of low-grade ore and shale or losing its identity entirely. Burchard[a] says of the grade of the ore:

> Its soft ore, now nearly all mined out either by surface trenches or drifts, is the best of the district. Its hard ore is also of high grade and has hitherto been for the most part held in reserve, since ore could be produced from the thicker Big seam at a lower cost per unit of iron.

The seam lies only a few feet below the Big seam and has a thickness of 4 to 6 feet.

Three samples from this seam were tested—two from the Ruffner mine and the third from the Hammond mine—they being from the middle part of the belt within which the seam has its maximum value.

SAMPLE 35. RUFFNER MINE.

Sample 35 represents the entire thickness of the Irondale seam in 1 slope, 18 left heading, 650 feet from the slope. The bed here is 4 to 4½ feet thick.

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[a] Burchard, E. F., and Butts, Charles, Iron ores, fuels, and fluxes of the Birmingham district, Alabama, with chapters on the origin of the ores, by E. C. Eckel: U. S. Geol. Survey Bull. 400, 1910, p. 47.

SAMPLE 36. RUFFNER MINE.

Sample 36 represents the entire thickness of the Irondale seam in 1 slope, 22 right heading, 450 feet from the slope. The seam here is 4 feet thick.

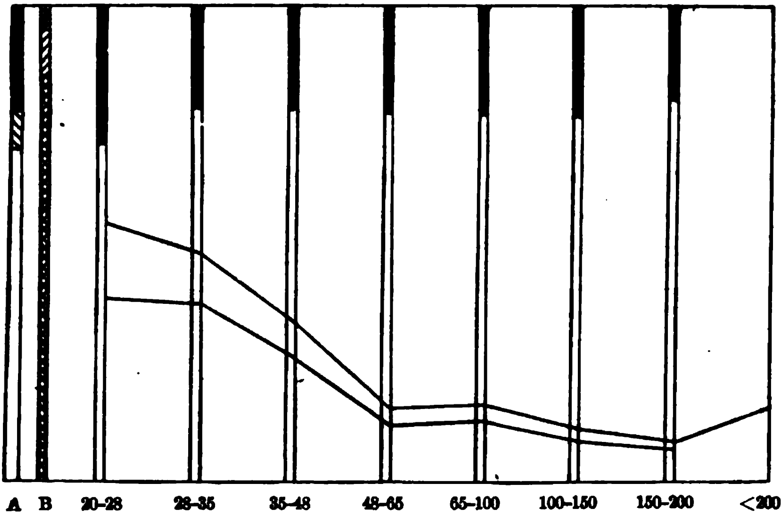

FIGURE 43.—Results of tests of sample 35.

SAMPLE 37. HAMMOND MINE.

Sample 37 was taken in the slope on the Irondale seam at the point where it is cut by the adit, that is, about 550 feet below the outcrop.

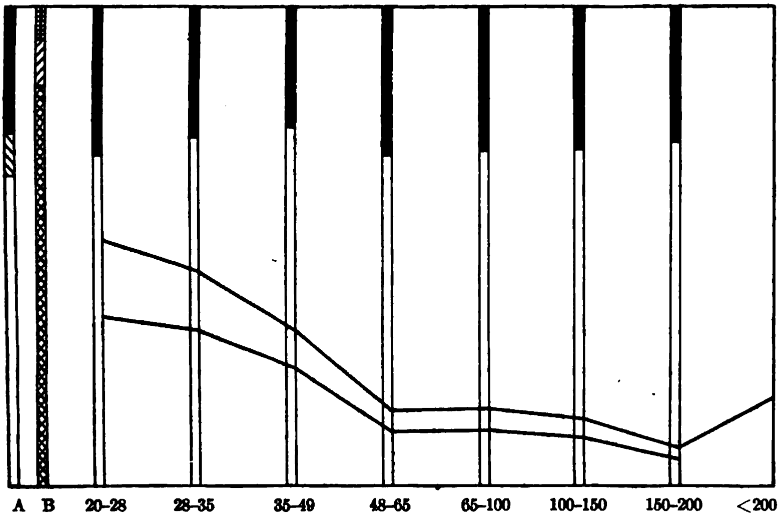

FIGURE 44.—Results of tests of sample 36.

The workable part of the bed was the lower 3½ feet, which was overlain by a thin layer of shale and 8 to 9 inches of low-grade ore discarded in mining.

COMMENTS ON TESTS OF ORE FROM IRONDALE SEAM.

Sample 37 is slightly better quality than the average ore of the lower bench of the Big seam. All three samples doubtless represent hard ore, and their average composition is 38 per cent Fe and 22.1 per cent SiO_2, or about the same as that of the lower bench Big seam ore. The concentrates average 45.1 per cent Fe and 14.2 per cent SiO_2, and hence are the same in iron, but 1.5 per cent higher in silica than the concentrates from the lower bench of the Big seam. A product composed of concentrates plus slimes show the same difference. However, the ratio of concentration averages a little less for the Irondale ores, so that the results obtained are practically the same as those obtained with the lower bench Big seam ores. Hence the tests indicate that the Irondale ores are amenable to any treatment that might prove feasible for those ores.

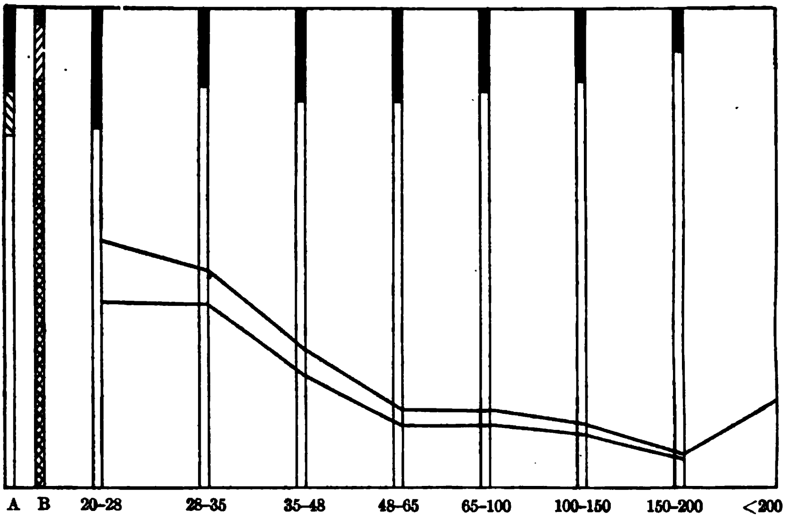

FIGURE 45.—Results of tests of sample 37.

ORE FROM THE IDA SEAM.

The following description of the Ida seam is given by Burchard:[a]

> This bed consists of 2 to 6 feet of rather siliceous ore associated with 14 to 16 feet of ferruginous sandstone. * * * Where worked, the bed is 3 to 5 feet thick, and soft ore only has been obtained from it in surface workings. Such ore carries 35 to 44 per cent of metallic iron, with a corresponding range in silica of 42 to 32 per cent.

Two samples from this seam were tested, one from the Ruffner mine and the other from the Hammond.

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[a] Burchard, E. F., and Butts, Charles, Iron ores, fuels, and fluxes of the Birmingham district, Alabama, with chapters on the origin of the ores, by E. C. Eckel: U. S. Geol. Survey Bull. 400, 1910, p. 46.

SAMPLE 38. RUFFNER MINE.

The outcrop of the Ida seam at the Ruffner mine is 5 feet thick, and the sample was taken at the face of an old open cut in what Mr. C. E. Barrett, the superintendent, called hard ore.

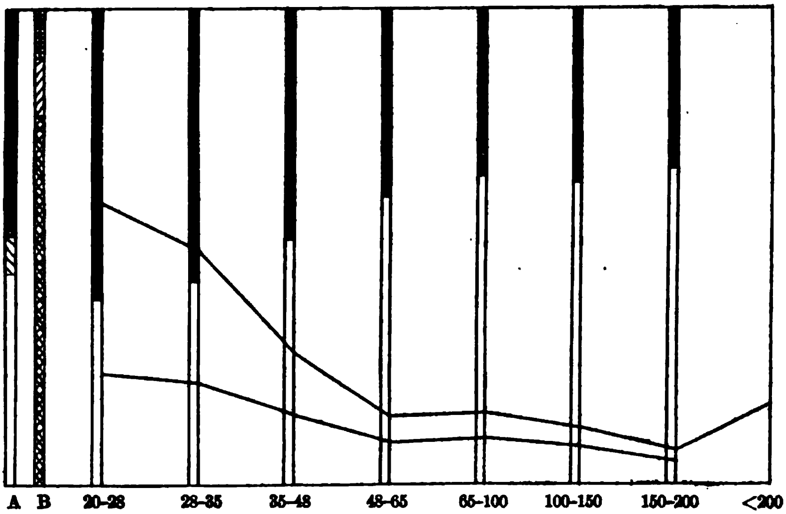

FIGURE 46.—Results of tests of sample 38.

SAMPLE 39. HAMMOND MINE.

Sample 39 was taken from the outcrop of the Ida seam along the face of the bed at the outside incline.

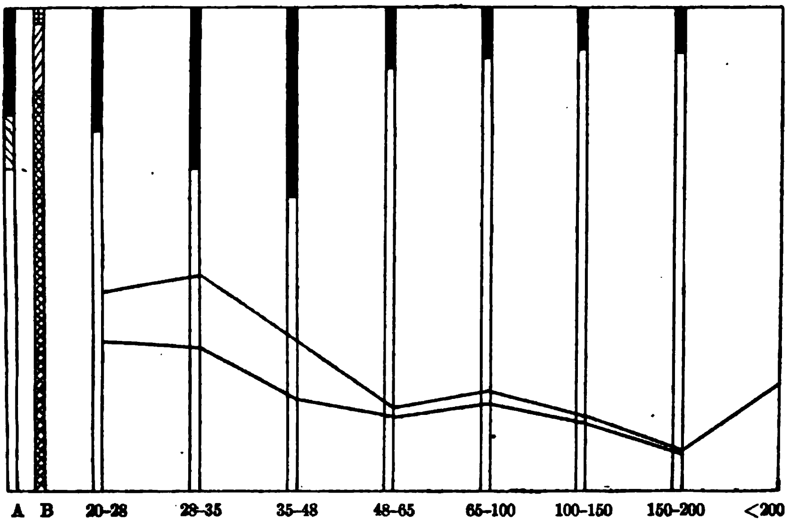

FIGURE 47.—Results of tests of sample 39.

COMMENTS ON TESTS OF ORE.

One sample represents hard ore, the other soft ore. The sample of soft ore is plainly finer grained than the sample of hard ore, as is shown by the screen analysis curves (figs. 46 and 47). The sample of hard ore (sample 38) is the poorest ore tested, carrying less than 30 per cent iron and more than 40 per cent silica, yet it yielded a concentrate carrying 47.5 per cent Fe and 14.7 per cent SiO_2, or just 2 per cent more iron and 2 per cent less silica than the average of the concentrates from the lower bench Big seam ore. This result would indicate that the siliceous Ida seam ores are as amenable to concentration as that ore.

The crude sample of soft ore (sample 39) runs high in iron, but its silica content is prohibitive. The concentrates from this sample show the high iron content obtained only in soft ore concentrates, and a silica content well within the allowable limit, and hence they can be advantageously used in the blast furnace.

RÉSUMÉ OF RESULTS.

There is a steady market in the Birmingham district for two types of iron ores—the calcareous hard red ores and the siliceous brown ores. The average composition of the hard red ores, as given by Phillips,[a] is 37 per cent Fe, 13.44 per cent SiO_2, 16.20 per cent CaO. The average of 23 samples of washed brown ore, as given by Burchard,[b] is 45 per cent Fe, 14.6 per cent SiO_2, 0.74 per cent Mn. Also there is available a limited quantity of workable soft ore, having an average composition, according to Phillips,[c] of 50.80 per cent Fe, 18.50 per cent SiO_2, and 1.20 per cent CaO. All of these ores average from 3 to 4 per cent Al_2O_3. As regards their smelting, the soft ores are in the same class as the brown ores, a higher iron content being offset by a higher silica content. The hard red ores grade by decrease of lime and increase of silica into siliceous ores, which, on account of their low iron content, can not compete with the brown ores, and hence are unworkable at present. Under favorable conditions, hard red ores of the average iron content can not be worked profitably if they carry about 8 per cent more silica than lime. By far the greater part of the red ore of the district falls below these requirements, and hence requires concentration to become available. As the concentrates obtained in the tests described herein are themselves siliceous and not self-fluxing, the brown ores serve as a basis for determining the value of the product obtained by concentration. Such a comparison follows:

Six samples of Big seam upper-bench ore taken underground were tested. Sample 4 from the Hammond mine, close to the surface, represents soft ore and includes the lower bench. Its concentrates run 51.7 per cent Fe and 18.4 per cent SiO_2; in other words, they equal the average workable soft ore, and their iron content is more than enough to carry the 4 per cent excess in silica as compared with the average brown ore. The crude ore is unworkable, with silica in excess of iron.

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[a] Phillips, W. B., Iron making in Alabama, 3d ed.: Geol. Survey of Alabama, 1912, p. 35.

[b] Burchard, E. F., and Butts, Charles, Iron ores, fuels, and fluxes of the Birmingham district, Alabama: U. S. Geol. Survey Bull. 400, 1910, p. 169.

[c] Phillips, W. B., work cited, p. 30.

Sample 1 from the Ishkooda mine is a workable, self-fluxing ore and was tested in order to ascertain the results of concentration. The concentrate is self-fluxing and 10 per cent higher in iron.

Sample 2 and 3 from the Spaulding mine represent upper bench ores that are workable only when iron is in good demand. Their concentrates are higher in iron than the average brown ores and, being nearly self-fluxing, are superior to those ores.

Two samples from the Ruffner mine, samples 5 and 6, represent extremely poor ore, with silica in excess of iron, averaging 28.8 per cent Fe and 30.9 per cent SiO_2. The concentrates are of much better quality, averaging 40.5 per cent Fe and 17.1 per cent SiO_2, but are too poor to be marketable.

These experiments show that the unworkable upper bench hematites of the Big seam will yield a concentrate of greater value than the brown ores unless the grade of the crude hematites is very low.

Four samples of ore were taken from the outcrop of the Big seam at the Frank White open cut. Two of these, samples 7 and 8, represent the upper 6 feet of the seam (all that was worked) but are so much lower in iron and higher in silica than the average soft ore that this part of the bed can be worked only when the iron trade is unusually active. Concentration yielded a high-grade product, carrying 53 per cent Fe and 12.9 per cent SiO_2. The middle 6 feet, sample 9, is of very low grade, with 5 per cent more silica than iron, yet the concentrate is only a little poorer than the average soft ore mined. Sample 10, from the lower 6 feet, shows about the same composition as the ore from the upper 6 feet, but the concentrate was poorer, having about the composition of the average workable soft ore, probably because of the finer grain. The entire seam averages 42.2 per cent Fe and 30.5 per cent SiO_2, and the concentrates from it 50.9 per cent Fe and 17.2 per cent SiO_2. Therefore, concentration at this mine could make a product of slightly better grade than the average of the soft ores out of an 18-foot seam of ore, of which only the upper 6 feet can even be considered as available in its crude state.

Twenty-four samples of Big seam lower bench ore were tested, of which one, sample 11 from the Ruffner mine, represents an extremely low-grade ore from the northeastern part of the district. The others represent the lower bench in that part of the district, where the upper bench is worked extensively and the lower bench is left unmined.

The sample of lower bench Ruffner ore is slightly poorer than the two samples of the upper bench ore tested, but the concentrate runs a little better. However, the concentrate is below the grade of the brown ores, and hence concentration would probably be unprofitable.

The 23 samples from Red Mountain between the Spaulding and Fossil mines show considerable uniformity in composition and in the results of concentration. This fact tends to emphasize the conclusions that can be based on the tests. The samples averaged 37.3 per cent Fe, 23.6 per cent SiO_2, and 8.9 per cent CaO; that is, their iron content is about the same as the average of the workable upper bench ores, but their silica content is 10 per cent higher and their lime content is more than 7 per cent lower. The concentrates obtained from these samples contain 0.4 per cent more iron and 2 per cent less silica than the brown ores, and, in addition, have a lime content of 8.1 per cent. Thus concentration yields a product of greater value than the brown ores. These results are the more encouraging because they were obtained with an iron recovery of 94.1 per cent and a concentration of 4 tons of crude ore into 3 tons of final product.

Three samples of ore from the Irondale seam were tested, samples 35 and 36 from the Ruffner mine, and sample 37 from the Hammond mine. The average iron and silica contents of these ores, 38 per cent Fe and 22.1 per cent SiO_2, is about the same as those of the lower bench Big seam ores. The concentrates average 45.1 per cent Fe and 14.2 per cent SiO_2, a little higher in silica than the Big seam lower bench concentrates, but still as good as the brown ores. In addition the concentrates doubtless have an appreciable lime content that would increase their value. On the whole, then, the ores tested from the Irondale seam have about the same composition as the Big seam lower bench ores from the producing part of the district, and are approximately as amenable to concentration.

Two samples of Ida seam ore were tested, one being hard ore and the other soft. Sample 37 from the Ruffner mine represents a poor hard ore, carrying 28.2 per cent Fe and 40.8 per cent SiO_2. The slimes from the test are so low grade that mixing them with the concentrates appreciably lowers the grade of the product; discarding them reduces the iron recovery to only 77.8 per cent, but the concentrate contains 47.5 per cent Fe and 14.7 per cent SiO_2, and is as low in silica as the average brown ore, with 2.5 per cent more iron. These results indicate that the ore has undergone partial alteration and is to be classed as semihard. The soft ore, sample 39 from the Hammond mine, is too poor to market in the crude state, but its concentrate had the same silica content as the brown ores and 10 per cent more iron.

With but few exceptions, then, and these being the unusually low-grade material, the experiments in concentrating the siliceous red ores have yielded final products that would have a value in excess of that possessed by the brown ores of the district.

The experiments above are based on crushing the ore to pass 20 mesh, probably as fine as would be practical. In order to determine, however, the degree to which the grade of the concentrate could be raised by still finer crushing, in five tests part of the ore was crushed to pass 35 mesh, and the concentrate resulting from the separation of both sizes was determined. For purposes of comparison the results are tabulated below.

Results of finer crushing.

Sample.	20-mesh test.					35-mesh test.					Slimes.	
	Concentrates.	Slimes.	Tailings.	Iron.	Silica.	Concentrates.	Slimes.	Tailings.	Iron.	Silica.	Iron.	Silica.
	P. ct.	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*
6	61.0	10.9	28.1	42.5	20.6	41.0	19.8	39.2	43.7	16.7	34.5	13.0
14	63.3	9.2	27.5	45.1	13.5	52.7	21.4	25.9	45.3	13.0	44.3	14.9
20	60.4	9.4	30.2	42.3	16.1	47.6	21.0	31.4	42.1	13.3	39.8	12.4
27	68.2	10.0	21.8	43.5	13.2	55.8	21.1	23.1	45.7	11.0	44.0	12.8
29	71.5	11.3	17.2	48.0	12.1	58.4	22.5	19.1	48.1	11.7	43.8	16.6
Average	64.9	10.2	25.0	44.3	15.1	51.1	21.2	27.7	45.0	13.1	....	....

These results show a very small average increase in iron in the finer concentrates and a 2 per cent decrease in silica. The amount of the tailings increased only a little, the change being almost entirely in the amount of the slimes at the expense of the concentrates. In the table the first and last slime analyses are of the 35-mesh slimes and the other three of the 20-mesh slimes. Thus increased sliming lowers the average grade of the slimes. If the slimes and concentrates are mixed to form a final product with as high iron recovery as for the similar product from the 20-mesh crushing, the grade of it will be but little if any higher and there will be no increase in grade of the final product to compensate for the finer crushing. Hence, 20-mesh crushing is fine enough to produce an adequate freeing of the constituent ore particles to yield an efficient separation.

In the above summary, except where otherwise indicated, the final product of concentration is considered to be the combined concentrate and slimes. The average composition of all the concentrates is 46.4 per cent Fe and 13.4 per cent SiO_2, and that of all the slimes is 44.7 per cent Fe and 13.5 per cent SiO_2. Of the total iron in the crude ore, that in the concentrates averages 80.6 per cent and that in the slimes 13.1 per cent. The average quantity of concentrates is 63.3 per cent, and that of slimes is 10.6 per cent. Consequently, if the slimes are added to the concentrates, the average composition of the final product will be 46.2 per cent Fe and 13.4 per cent SiO_2; in other words, this procedure does not change the silica content and lowers the iron content only 0.2 per cent, but the average iron recovery is raised 13.1 per cent, or from 80.6 per cent to 93.7 per cent. The advantages of mixing these two products in raising the iron recovery and eliminating the need of slime treatment are obvious.

That crushing alone brings about a concentration of the iron in the finer sizes and a concentration of the silica in the coarser sizes is strikingly shown, the slimes (less than 200 mesh) from the crude ore being of practically the same composition as the concentrates from the sizes coarser than 200 mesh. In order to show this tendency in more detail the concentrates and tailings of each mesh size of five samples were analyzed, and from these analyses the composition of the crude ore of each of these sizes was calculated. The results are tabulated below.

Increase of iron in the slimes.

	Sample 3.		Sample 8.		Sample 10.		Sample 19.		Sample 39.		Average.	
	Fe.	SiO_2.	Fe.	SiO_2.	Fe.	SiO_2.	Fe.	SiO_2.	Fe.	SiO_2.	Fe.	SiO_2.
	P.ct.	*P.ct.*	*P.ct.*	*P.ct.*	*P.ct.*	*P.ct.*	*P.ct.*	*P.ct.*	*P.ct.*	*P.ct.*	*P.ct.*	*P.ct.*
Crude ore......	32.4	29.3	35.9	27.0	35.7	15.0	38.4	20.2	39.5	19.3	36.4	22.2
20 to 28........	28.8	40.1	29.4	38.5	32.5	20.5	32.1	32.3	31.9	29.9	30.9	32.3
28 to 35........	29.6	33.1	32.2	33.0	36.1	15.9	36.6	23.8	35.2	24.4	33.9	26.0
35 to 48........	31.8	24.7	36.1	24.0	37.1	14.0	37.4	19.7	37.3	19.9	35.9	20.5
48 to 65........	32.5	20.1	39.5	19.3	35.0	12.4	38.6	16.0	38.3	17.3	36.8	17.0
65 to 100.......	33.9	18.3	40.9	18.7	35.3	12.2	39.9	14.8	38.7	16.2	37.7	16.0
100 to 150......	36.3	16.7	43.5	16.4	38.4	10.9	42.3	13.2	43.0	15.2	40.7	14.5
150 to 200......	43.3	12.7	45.1	16.0	42.3	9.7	44.7	13.3	45.3	14.6	44.1	13.3
200.............	39.8	12.7	46.1	14.0	40.0	8.9	45.5	11.3	47.4	11.7	43.8	11.7
Concentrate....	42.3	16.1	45.5	11.9	46.2	7.9	45.3	12.0	45.3	11.8	44.9	11.9

This table shows that the two coarsest sizes, 20 to 28 mesh and 28 to 35 mesh, are of lower grade than the crude ore, whereas the finer sizes are of increasingly higher grade.

PRACTICAL VALUE OF THE EXPERIMENTS.

The bearing of the results of these experiments on probable results of concentration on a commercial scale by wet methods or with magnetic separation will now be considered. The author knows that the method of concentration he used is not feasible on a commercial scale, and its efficiency of separation is not obtainable in a commercial plant. The experiments were made to determine the degree to which the silica might be separated from the iron—that is, granted a perfect separation of the mineral particles, what is the result?

If this result indicates that concentration is feasible, the devising of a commercial method of separation would be encouraged. If, on the other hand, a perfect separation of the mineral particles does not yield a marketable product of sufficient value, there is no use working at separation on a commercial scale. The results obtained in the experiments have demonstrated that, so far as the experiments go, a solution of the problem is possible.

Results obtained in the application of the Moxham-du Pont haloid process to the concentration of low-grade iron ores open the possibility of the commercial application of the method used in this investigation and may render the results of these experiments directly applicable. But while this process is in the experimental stage, it will be better to consider the bearing of these results on separation by ordinary wet methods or by magnetic methods.

Wet methods of separation are based essentially on the differences in specific gravity of the mineral components of an ore. In the author's experiments the ores were crushed to pass 20 mesh, a fineness that is not at all unusual in ore mills. As the iron oxide in the ore tested is much softer than the silica that is to be removed, crushing causes an increase of iron in the finer sizes. So marked is this concentration of the iron that the crude material passing through 200 mesh has practically the same iron content as that obtained by concentrating the material coarser than 200 mesh. This fact is of great importance as it eliminates the slime problem in the use of wet methods. The material finer than 200 mesh may be separated by washing immediately after crushing, allowed to settle, and then added to the concentrate obtained from the rest of the material. The concentrates obtained with the same ratio of concentration will not be of as high grade as those made in the experiments because of less complete separation, but this difficulty could be met by increasing the ratio of concentration a little, thus raising the grade of the concentrate and lowering the iron recovery. As the concentrates from the tests of lower bench ores of the Big seam, for example, represented an iron recovery of 94.1 per cent, it is evident that a little lowering of the iron recovery to meet the decreased efficiency of commercial work would still permit a good recovery and give a concentrate of high enough grade. Hence there seems to be no reason to doubt the possibility of obtaining equally good concentrates, although with a lower iron recovery, on a commercial scale by wet methods.

Equally favorable conclusions can be drawn in regard to the results that may reasonably be expected from applying magnetic separation to remove the more magnetic particles of the crushed ore, as in an ore similar to that under discussion the more magnetic particles are those high in iron and low in silica. In other words, the magnetic susceptibility is determined by the same factors as the specific gravity and is a linear function of the latter. Consequently, crushing frees the ore particles for magnetic separation to exactly the same degree as for specific gravity separation. Therefore a perfect magnetic separation should yield results identical with those obtained by a perfect specific gravity separation. But in ordinary practice a perfect magnetic separation is no more obtainable than a perfect gravity separation. In either case the grade of the final product can be maintained by increasing the ratio of concentration a little at the expense of the iron recovery. As stated above, in the author's concentration experiments iron recovery was so high that a small decrease in it would not be prohibitive.

The experiments have, consequently, demonstrated the possibility of producing from ores now valueless and being permanently lost by present mining methods a marketable product. Also, it is probable that as good results can be obtained commercially by any of the usual methods of ore treatment if the decrease of efficiency is compensated by increasing the ratio of concentration.

COMMERCIAL ASPECTS OF THE PROBLEM.

As the tests indicate that concentrates of higher grade than the best of the ores now available in the Birmingham district can be obtained from the siliceous ores of the district, the question remains whether concentration would be profitable. The answer requires the determination of two things—first, the cost of crushing, concentrating, and nodulizing the concentrates; second, the value of the concentrates as compared with the crude ore. The author does not have the necessary data and does not consider himself competent to supply such figures, for they should be based on experience with concentration on a large scale and on familiarity with mining costs and the price of ore in the district. However, some general suggestions are offered.

Beneficiation of low-grade iron ores is widely practiced; it includes magnetic separation and nodulizing of low-grade magnetites. Magnetic concentration of the siliceous red hematites would involve higher treatment cost than for magnetites because, generally speaking, finer crushing would be needed. On the other hand, these ores can be mined cheaper than many magnetites thus treated. Gravity methods of wet concentration for low-grade iron ores have been successfully used on a large scale in the Lake Superior region, and have made available great quantities of ore that are too low in grade to be shipped crude. Hence it is reasonable to presume that the costs of treatment would not necessarily be prohibitive.

The margin allowable for treatment costs can be determined more nearly. During the years 1910 to 1915 the red ores and the brown ores of the district showed the following range in prices, according to the United States Geological Survey:[a]

---

[a] Figures for 1910 and 1911 from Mineral Resources of the United States for 1911, Part 1, p. 130. Figures for 1912 and 1913 from Mineral Resources of the United States for 1913, Part I, p. 305. Figures for 1914 and 1915 are from Mineral Resources of the United States for 1915, Part 1, p. 291.

Prices of red and brown ores of Birmingham district.

	1910	1911	1912	1913	1914	1915	Average.
Red ores	$1.19	$1.17	$1.16	$1.18	$1.21	$1.25	$1.19
Brown ores	1.53	1.47	1.43	1.61	1.49	1.68	1.54

During these six years the average price of the brown ores was 35 cents in excess of that of the red ores. It has been seen that the value of the concentrates would exceed that of the brown ores, and the cost of mining the lower bench ores in mines working the upper bench ought to be less than the present cost of mining the upper bench alone. Therefore, the margin allowable for treatment should be in excess of 35 cents. How much larger it might be can be determined by those having complete data on mining costs and the value of the ores as determined by chemical composition.

That one must guard against assuming too low a cost for mining lower bench as compared with upper bench ores is pointed out by Burchard, who says:[a]

> To recover the lower ore at present would, however, introduce serious complications in mining. At the Tennessee company's mines the slopes are cut in the lower bench of the seam about 8 feet below the working level of the headings, so that tramcars of ore may be dumped over the tipple into the skip in the slope. If the lower bench of ore were mined also, the slopes would have to be depressed 8 feet below the bottom of this bench in order to accommodate skip haulage. This would necessitate maintaining for some distance a slope height of 25 to 28 feet, which would be excessive and would involve deepening and retimbering the present slopes. Beyond the present faces the slopes might be driven below the parting, leaving the upper bench above the slope. The matter of setting props to support the roof in the workings where 18 to 20 feet of ore are taken out would also prove inconvenient.
>
> In slopes where tram haulage is employed the problem is simpler, since it is not necessary to depress the slope below the bottom of the ore that is being mined. Part of the ore from the lower bench can be obtained in either case by cutting the floors of the rooms lower. By this method, however, the floors become too flat to permit the ore to be shot down to the cars, and the expense of moving it is thereby increased.
>
> Evidently the problem is discouraging in view of present costs and conditions, but if for every ton of ore mined from the lower bench nearly a ton of ore in the upper bench might be saved for future use, it would seem worth while to carry the investigation as far as possible, even to the extent of equipping one slope to mine the whole seam on a scale that would enable the relative costs to be compared on a working basis.

In spite of such difficulties there certainly ought to be a margin in favor of the lower bench ore. As those ores are not utilized at present and are being destroyed as reserves, and as the mines are now equipped for mining the upper bench ore, none of the charges now assumed by the upper bench ore should be distributed between it and the cost of producing the lower bench ore. The cost of mining both ores should be determined, and then from that cost should be subtracted the present cost of mining the upper bench ore. The difference is all that should fairly be charged against the lower bench costs and would certainly be less than the present figures for the upper bench ores. The difference would be applicable to meeting concentration charges. Moreover, a decidedly important consideration would be the doubling of the ore reserves.

---

[a] Burchard, E. F., and Butts, Charles, Iron ores, fuels, and fluxes of the Birmingham district, Ala.: U. S. Geol. Survey Bull. 400, 1910, pp. 124, 125.

From this consideration of the factors involved, it seems reasonable to assume that the margin allowable for meeting concentration charges is considerably more than 35 cents.

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MOUNTAIN FROM WHICH

Bulletin 111 Mineral Technology 15

DEPARTMENT OF THE INTERIOR
FRANKLIN K. LANE, SECRETARY
BUREAU OF MINES
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MOLYBDENUM; ITS ORES AND THEIR CONCENTRATION

WITH A DISCUSSION OF

MARKETS, PRICES, AND USES

BY

FREDERICK W. HORTON

WASHINGTON
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MOLYBDENUM; ITS ORES AND THEIR CONCENTRATION.

By **Frederick W. Horton.**

INTRODUCTION.

With large deposits of low-grade molybdenum ore and a latent market for molybdenum, which with development might perhaps equal that for tungsten, the United States has for years made no production of molybdenum ore worthy of mention. In fact, during the decade previous to 1914, there was no output of molybdenum ore in this country except in 1905, 1906, and 1907, when a few small lots of molybdenite and wulfenite were marketed.[a]

To ascertain why this possible source of economic wealth should remain undeveloped, the United States Bureau of Mines undertook an investigation in 1914, with a view of assisting the establishment of a molybdenum industry in this country. A preliminary review of the situation showed the following conditions:

Briefly there was a lack of any considerable demand for molybdenum for the one known use that might consume any large tonnage of it, that is, for the manufacture to certain alloy steels to which molybdenum imparts properties roughly similar to those produced by tungsten. It was at once evident that the market for molybdenum in alloy steels must be developed, or that extensive new uses for the metal must be created, before the demand would be sufficient to warrant any extensive mining of molybdenum ore.

Investigation showed that one of the chief factors in retarding the development of any demand for molybdenum by the alloy-steel trade was that manufacturers who might use, or might investigate the possibilities of using, the metal were kept out of the market by the fear of not being able to obtain steady supplies. On the other hand, those who might be interested in the development of some of the extensive low-grade molybdenite or wulfenite deposits in this country were prevented from doing so by the small visible demand and the fear that any large production would glut the market. Owing to these conditions the mining of molybdenum has in the past been confined almost entirely to small-scale operations on high-grade streaks of molybdenite ore, and the methods of recovery have been limited largely to cobbing and hand picking.

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[a] Mineral Resources U. S. for 1904 to 1913, U. S. Geol. Survey, 1905 to 1914.

SCOPE AND PURPOSE OF INVESTIGATION.

The problem before the Bureau of Mines was to ascertain the character and extent of the deposits of molybdenum ore in the United States from which supplies requisite for the development of the market might be obtained, and how the ores might best be concentrated into a marketable product. These features were the ones that the investigation described in the following pages principally covered.

The direct purpose of this bulletin is, on the one hand, to prove to possible consumers of molybdenum that the element is not as rare as commonly supposed, and that this country possesses many deposits of low-grade ore from which large supplies may be derived, and on the other hand, to prove to present and prospective producers of molybdenum that there is a latent market for their product in the alloy-steel trade, which needs only the assurance of steady supplies for a considerable development. It is also intended to assist the miner of molybdenum by giving him information regarding various methods of concentration that have proven applicable to the different types of molybdenum ore, and by acquainting him with market conditions, uses, prices, etc. The various molybdenum minerals are described in detail, and tests for their determination are given to aid the prospector in recognizing them. In addition to the most applicable and reliable qualitative tests for molybdenum, the quantitative determination of the element is described in some detail, as the methods of analyses recommended in the ordinary textbooks will not in general give correct results, particularly on low-grade ores.

PRESENT CONDITION OF THE MOLYBDENUM INDUSTRY.

The unprecedented demand for steel-hardening metals occasioned by the European war is slowly drawing the attention both of prospective consumers and of producers to possibilities in regard to molybdenum. Several mining operations looking toward a considerable production of both molybdenite and wulfenite have already been commenced, and manufacturers are investigating the possibilities of using molybdenum in a large way. The fact that molybdenum is now (April 1, 1916) selling at $3 to $4 a pound, as compared with $8 to $10 a pound for tungsten, and the further fact that 1 pound of molybdenum will produce approximately the same results as 2 or 3 pounds of tungsten, are serving to quicken this interest.

Under the influence of the high prices being paid for the metal and the unusual demand, the whole molybdenum industry is developing as never before, not only in the United States, but also in Queensland, New South Wales, and Norway, the three countries that have previously supplied practically the entire world's production. In this country, since late in 1914, a fair-sized molybdenite mine has been opened up on a low-grade deposit at Red Mountain, near Empire, Colo., and extensive shipments of ore have been made. Many molybdenite mines and prospects throughout the West have produced small lots of high-grade ore, and some of them have accumulated dumps of milling ore. A noteworthy amount of development work has been done at many of these properties, and several of them are rapidly being put into condition to become producers. Since the summer of 1914 a considerable tonnage of wulfenite concentrates of medium grade has been derived by milling operations on some large tailing piles at Mammoth, Ariz., and in the spring of 1915 the output from this source was more than doubled. It is lately reported that a mill has been installed to treat wulfenite ore from the Old Yuma mine, near Tucson, Ariz.

Developments have already progressed to such an extent that, in the author's opinion, the molybdenum production of the United States in 1915 will compare favorably with that of Queensland, New South Wales, or of Norway, and it may even exceed them.

THE FUTURE OF THE MOLYBDENUM INDUSTRY.

Of course, the extremely high prices that are now being paid for molybdenum can not last, but even under normal conditions the author sees no reason why molybdenum should not compete with tungsten in the manufacture of many alloy steels. It is surely as plentiful an element as tungsten, its ores can be mined as cheaply as those of the latter metal, and although high-grade concentrates from molybdenite ores will probably cost a little more to produce than from tungsten ores, the fact that much less molybdenum need be used to produce a given result should give it the advantage in the market. If steady supplies of molybdenum can be obtained at or near the prices at which tungsten is offered, the demand for it can not help but develop greatly within a few years.

ACKNOWLEDGMENTS.

The writer takes pleasure in acknowledging his indebtedness to Mr. Karl L. Kithil, mineral technologist of the Bureau of Mines, for his kindly advice in the preparation of this report, and to Dr. Charles L. Parsons, chief of the division of mineral technology of the bureau, for many valuable suggestions. The author's thanks are also extended to Messrs. R. B. Moore, J. C. Morgan, and H. A. Doerner, of the Denver office of the bureau, for chemical analyses, and to Dr.

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The writer expresses his appreciation to the following companies and individuals for their kindness in placing at his disposal the equipment of their testing laboratories for concentration work: W. G. Swart, of the American Zinc Ore Separating Co.; Henry E. Wood, president of Henry E. Wood & Co.; Albert M. Plumb, of the Plumb Jig Co.; Frank E. Shepard, president of the Denver Engineering Works Co.; and W. S. Myers, president of the Sutton, Steele & Steele Manufacturing, Mining & Milling Co., all of Denver, Colo. Acknowledgments are also made to the following individuals and companies for their courtesy in supplying the writer with molybdenum ores for concentration tests: George H. Daily, Tucson, Ariz.; R. J. A. Widmar, Breckenridge, Colo.; William Young and C. S. Lively, Empire, Colo.; H. Leal, Denver, Colo.; Smith & Sawyer, Kingman, Ariz.; Young Bros., Shultz, Ariz.; Santa Maria Molybdenum Mining & Milling Co., San Diego, Cal.; and the Grand View Mining & Development Co., Twin Bridges, Mont.

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PART I. THE MOLYBDENUM MINERALS.

The following table lists the known molybdenum minerals and gives their theoretical composition, and the percentage of molybdenum each mineral contains as based on this composition:

Molybdenum minerals.

Mineral.	Composition.	Per cent molybdenum.	Mineral.	Composition.	Per cent molybdenum.
Molybdenite [a]	MoS_2	59.95	Powellite	$CaMoO_4$	47.98
Wulfenite	$PbMoO_4$	26.15	Pateraite	$CoMoO_4$	43.84
Molybdite	$Fe_2O_33MoO_37\frac{1}{2}H_2O$	39.63	Achrematite	$3(3Pb_3As_2O_8PbCl_2)4=(Pb_2MoO_5)$.	3.40
Ilsemannite [b]	MoO_24MoO_3(?)	68.18			
Belonesite	$MgMoO_4$	52.08	Eosite	Vanado-molybdate of lead.	....

[a] An amorphous variety of molybdenite called jordisite is said to exist (see p. 9.)
[b] Composition doubtful (see p. 12).

Of the above list only the first two minerals, molybdenite and wulfenite, have thus far proved of commercial importance. With the exception of molybdite, the other minerals listed are rare and are to be considered only in the light of interesting mineralogical occurrences. Besides these there are other molybdenum minerals that have been reported, but information concerning them is so incomplete both as regards occurrence and composition that their existence as definite species can not be considered as authenticated. (See p. 12.)

MOLYBDENITE.

Molybdenite is the disulphide of molybdenum (MoS_2) and contains 59.95 per cent of molybdenum and 40.05 per cent of sulphur. It is a soft, opaque, lead-gray mineral with a metallic luster and greasy feel. It commonly occurs in flakes or scales having a prominent basal cleavage and resembling some micas in the way the flakes may be split into thin leaves. Finely granular and massive forms are also common. The mineral is sectile and in the flaky form the laminæ are flexible but not elastic. In hardness it ranges from 1 to 1.5, being so soft that it soils the fingers readily in handling and marks paper, on which it leaves a bluish-gray trace. On porcelain its streak is slightly greenish. Its specific gravity has been variously determined at 4.7 to 4.8.[c]

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[c] Dana, E. S., A system of mineralogy, 6th ed., 1911, p. 41.

On account of many similar characteristics molybdenite is often confused with graphite, but it may be easily distinguished from the latter as graphite has a much lower specific gravity, 2.09 to 2.23, and its streak is lead-gray on both paper and porcelain. Heating a fragment of the mineral in a closed tube will conclusively settle any further question as to its identity, as the strong sulphurous odor given off by molybdenite is entirely lacking with graphite.

Molybdenite crystallizes in hexagonal form and the crystals are tabular or short and slightly tapering prisms. The prismatic planes are horizontally striated and on the base of some crystals there are striae normal to the edges. In Plate I, which illustrates characteristic forms of molybdenite crystals, the horizontal striæ on the prismatic faces may readily be seen. Many molybdenite aggregates exhibit a distinct radial formation. This typical structure is well shown in Plate II, *A*. A typical molybdenite ore is shown in Plate III, *A*.

An artificial crystallized molybdenite with a specific gravity of 5.06 has been made by melting together potassium carbonate, sulphur, and molybdic oxide in a platinum crucible.[a]

Molybdenite commonly alters to molybdite, but it is also reported as sometimes altering to ilsemannite. (See pp. 12 and 13.)

TESTS FOR MOLYBDENITE.

Different methods of testing molydenite follow:[b] When heated in an open tube molybdenite gives off sulphurous fumes, and a pale yellow crystalline sublimate of molybdenum trioxide (MoO_3) is formed. Before the blowpipe, the mineral is infusible and imparts a yellowish-green color to the flame. On charcoal in the oxidizing flame, pulverized molybdenite gives a strong odor of sulphur and the charcoal is coated with crystals of molybdic oxide which appears yellow while hot and white when cold. Near the heated mineral the coating is copper-red, and if the white coating is touched with an intermittent reducing flame, it becomes a beautiful azure blue. Molybdenite is decomposed by nitric acid, leaving a white or grayish residue (molybdic oxide).

OCCURRENCES OF MOLYBDENITE.

Probably three-fourths of the reported occurrences of molybdenite are in acid igneous rocks such as granites, pegmatites, trachytes, and syenites. Next in order of importance as regards deposits of the mineral are the metamorphic rocks, molybdenite being found in serpentines and gneisses, and in amphibolite, chlorite, talc, and mica schists, etc. Next follow the sedimentary rocks with occur-

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[a] Dana, E. S., op. cit., p. 42.

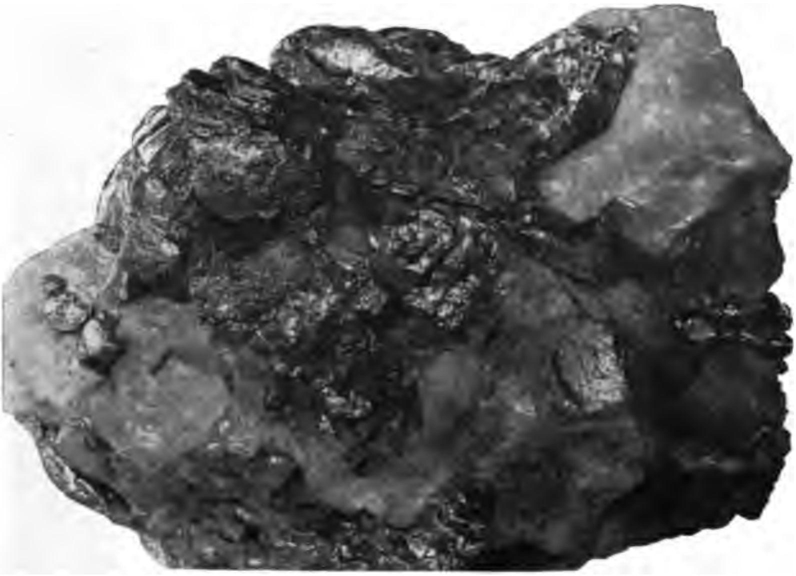
[b] Dana, E. S., op. cit., p. 41.

A. MOLYBDENITE CRYSTAL FROM CROWN POINT MINE, RAILROAD CREEK, CHELAN COUNTY, WASH. (TWO-THIRDS NATURAL SIZE.)

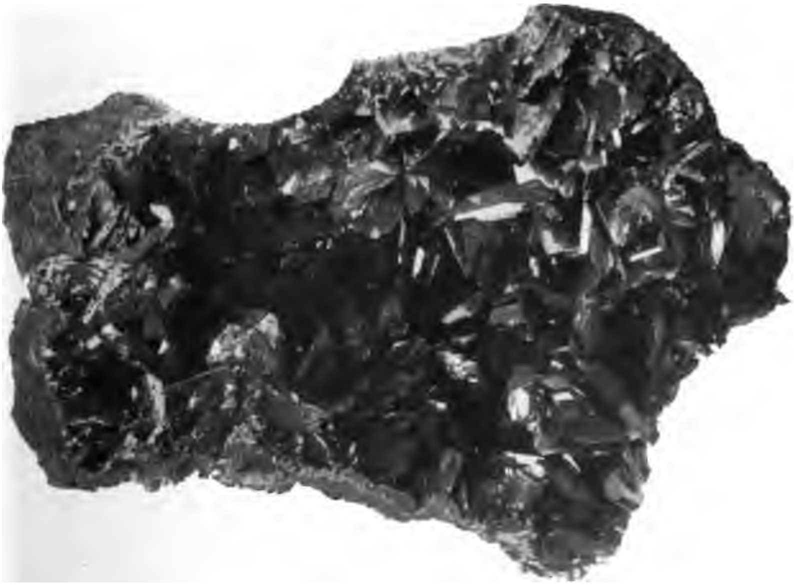
B. ANOTHER MOLYBDENITE CRYSTAL FROM CROWN POINT MINE. (ONE AND ONE-HALF TIMES NATURAL SIZE.)

A. MOLYBDENITE CRYSTAL FROM NEAR BUENA VISTA, COLO. (ONE AND ONE-HALF TIMES NATURAL SIZE.)

B. FLAKE FROM MOLYBDENITE CRYSTAL SHOWING ALTERATION TO MOLYBDITE, FROM SANTA MARIA PROPERTY, SAN DIEGO COUNTY, CAL. (FOUR TIMES NATURAL SIZE.)



A. MOLYBDENITE ON QUARTZ FROM CROWN POINT MINE, RAILROAD CREEK, CHELAN COUNTY, WASH. (THREE-FIFTHS NATURAL SIZE.)



B. WULFENITE CRYSTALS LINING GEODE FROM MAMMOTH MINE, PINAL COUNTY, ARIZ. (ONE-HALF NATURAL SIZE.)

rences of the mineral in conglomerates and limestones, and finally the basic igneous rocks with a few deposits in gabbros, basalts, etc.

Localities in the United States where molybdenite has been reported are named in part 2 of this report, and detailed descriptions of many characteristic occurrences are given there.

ACCOMPANYING MINERALS.

Besides the occurrence of gold and of various silver minerals with molybdenite, the author has noted the following accompanying minerals: Molybdite, bornite, chalcopyrite, malachite, azurite, chrysocolla, native copper, pyrite, galena, pyrrhotite, magnetite, limonite, sphalerite, bismuthinite, cassiterite, wolframite, scheelite, calcite, quartz, orthoclase, pyroxene, hornblende, garnet, epidote, tourmaline, muscovite, and biotite. The following additional minerals have been recorded by Crook[a] as associated with molybdenite: Arsenopyrite, fluorite, barite, apatite, rutile, oligoclase, tremolite, scapolite, and zircon; and Umpleby[b] has noted tetrahedrite, chalcocite, cuprite, hubnerite, and manganese oxides as accompanying minerals. Dolomite, talc, beryl, phlogophite, and löllingite are also mentioned by Hintze[c] as occuring with molybdenite.

JORDISITE.

According to Cornu,[d] there exists a black powdery form of molybdenum sulphide that he considers to be distinct from crystalline molybdenite. It is described as "colloidal molybdenum sulphide," and is said to alter to ilsemannite[e] and to occur at Himmelfurst, Freiberg, Saxony. Jordisite is listed as a variety of molybdenite by Cahen and Wootton.[e]

WULFENITE.

Wulfenite is a molybdate of lead ($PbMoO_4$) and theoretically contains 26.15 per cent of molybdenum and 56.42 per cent of lead. It is a heavy, brittle, subtransparent to subtranslucent mineral with a resinous or adamantine luster and is generally of a wax or orange-yellow color. It may, however, be siskin and olive green, yellowish-gray, brown, grayish-white to nearly colorless, or orange to bright red. Its hardness is 2.75 to 3 and its specific gravity is 6.7 to 7. It has a subconchoidal fracture and a white streak. In planes parallel with its crystal pyramid faces it has a smooth cleavage, but in other directions the cleavage is less distinct. It crystallizes in the tetragonal system with pyramidal hemihedrism. The crystals are commonly square and tabular and are sometimes extremely thin, with a vicinal pyramid replacing the basal plane. Less frequently the crystals are octahedral or prismatic, the prismatic faces showing the hemihedrism characteristic of the mineral. Plates III, *B*, and IV, *A*, illustrate common tabular forms of wulfenite crystals and Plate V, *A*, shows a fine specimen illustrating the extremely thin crystals referred to above. Wulfenite crystals of mineral form are illustrated in Plate IV, *B*.

---

[a] Crook, A. R., Bull. Geol. Soc. America, vol. 15, 1904, pp. 283-288.

[b] Umpleby, J. B., Geology and ore deposits of Lemhi County, Idaho: U. S. Geol. Survey Bull. 528, 1913, p. 73.

[c] Hintze, Carl, Handbuch der Mineralogie, Bd. 1, pp. 410-418.

[d] Cornu, F., Natürliches kolloides Molybdänsulfid (Jordisit): Ztschr. Chem. Ind. Kolloide, Bd. 4, 1909, p. 190.

[e] Cahen, Edward, and Wootton, W. O., The mineralogy of the rarer metals; a handbook for prospectors. 1912, pp. 51-52.

Wulfenite generally occurs in well-crystallized forms, but it is also found in coarse-grained or fine-grained masses. Small percentages of calcium, chromium, vanadium, copper, iron, or aluminum are generally present in wulfenite as impurities.

Among others, the following characteristic analyses of American wulfenites are given by Dana: [a]

Characteristic analyses of American wulfenites.

Source of sample.	Per cent MoO_3.	Per cent PbO.	Per cent other oxides.	Total per cent.
Eureka County, Nev.	39.33	61.11	1.04 CaO_3, 0.38 Fe_2O_3	101.86
Phoenixville, Pa	39.21	60.00	.38 Cr_2O_3	99.59
Do	37.47	60.30	1.28 V_2O_5	99.05

TESTS.[b]

Before the blowpipe wulfenite decrepitates and fuses below 2. With borax in the oxidizing flame it gives a colorless glass which in the reducing flame becomes an opaque black or dirty green with black flocks. With salt of phosphorus in the oxidizing flame wulfenite gives a yellowish-green glass which becomes dark green in the reducing flame. The powdered mineral, when heated with soda on charcoal, yields metallic lead. On evaporation with hydrochloric acid wulfenite is decomposed, forming lead chloride and molybdic oxide. Moistening the residue with water and adding metallic zinc gives an intense blue color which persists after dilution.

OCCURRENCE AND ACCOMPANYING MINERALS.

Deposits of wulfenite are confined almost wholly to veins, in which it occurs associated with other lead minerals. Localities in the United States where wulfenite has been reported are named in part 2, and detailed descriptions of several characteristic occurrences are also given.

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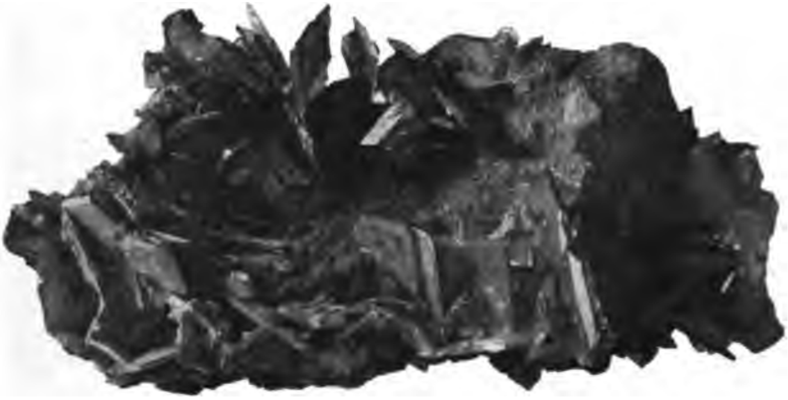
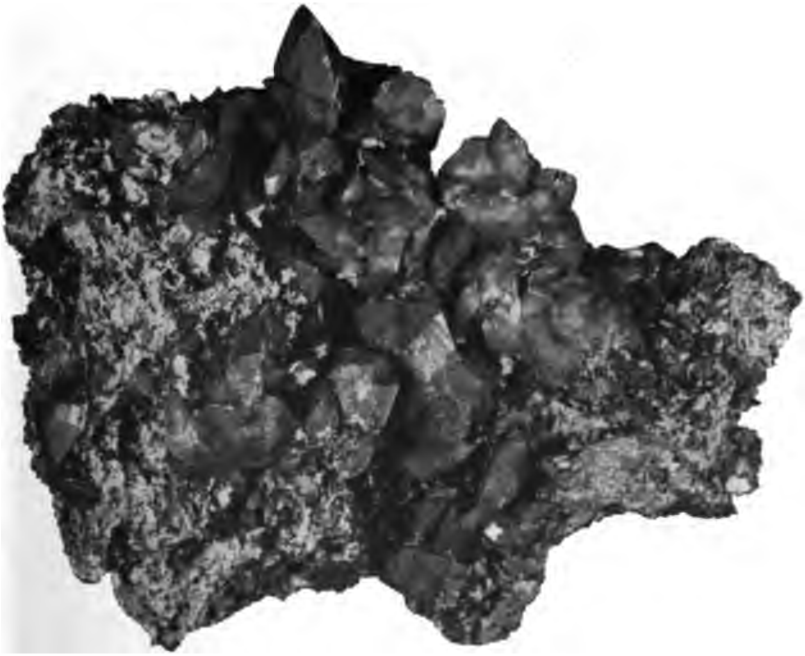
[a] Dana, E. S., A system of mineralogy, 6th ed., 1911, p. 991.

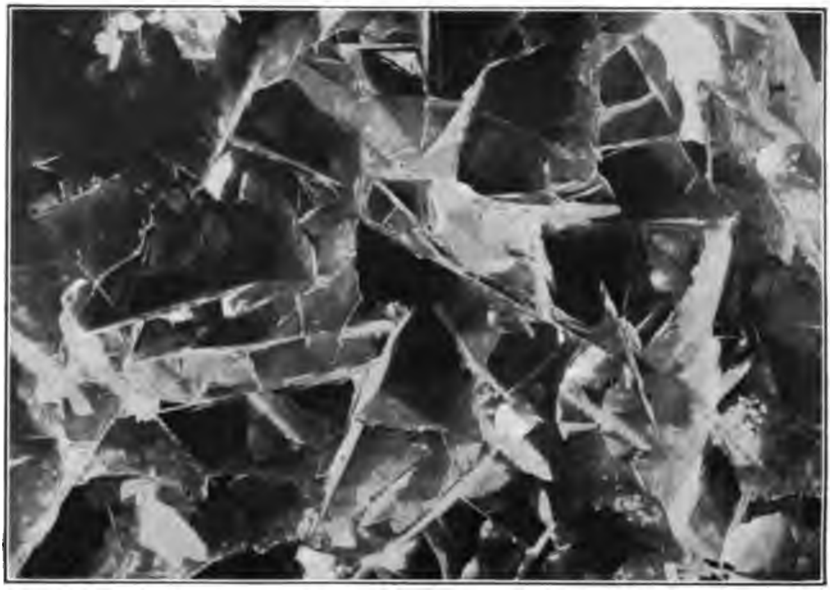
[b] Dana, E. S., loc. cit.



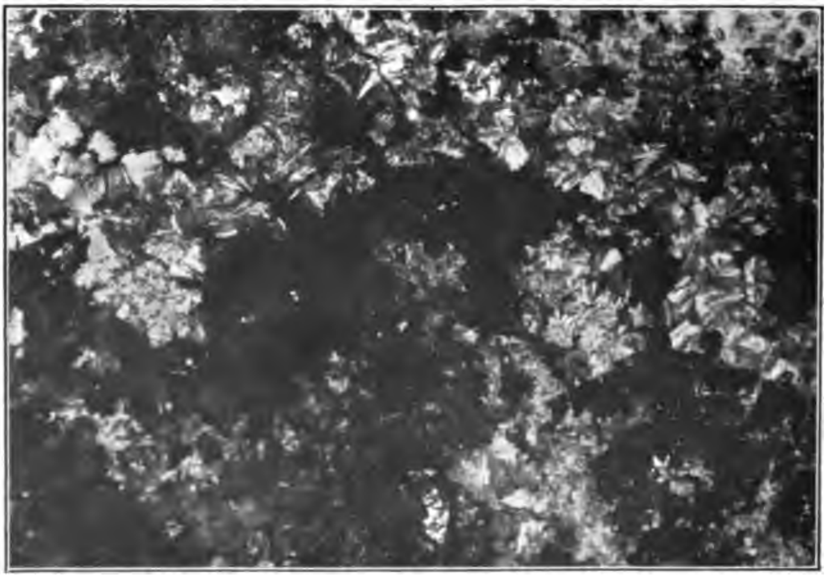
A. WULFENITE CRYSTALS FROM YUMA COUNTY, ARIZ. (NATURAL SIZE.)



B. WULFENITE CRYSTALS FROM LUCKY BILL MINE, NEAR SANTA RITA, N. MEX., SHOWING UNUSUAL FORMS. (ONE AND ONE-FOURTH TIMES NATURAL SIZE.)



A. WULFENITE CRYSTALS FROM EUREKA, NEV. (NATURAL SIZE.)



B. MOLYBDITE CRYSTALS ON IRON-STAINED GRANITE FROM SANTA MARIA, SAN DIEGO COUNTY, CAL. (THREE TIMES NATURAL SIZE.)

Outside of the gangue-forming minerals, wulfenite has been found associated with gold and various silver minerals, with galena, cerussite, pyromorphite, vanadinite, anglesite, and descloizite. In connection with the occurrence of gold with wulfenite, it may be of interest to note that the native gold is sometimes contained directly in the wulfenite crystals themselves. (See p. 115.)

MOLYBDITE.

Molybdite is a hydrous ferric molybdate ($Fe_2O_3.3MoO_3.7\frac{1}{2}H_2O$) and theoretically contains 39.63 per cent of molybdenum. It is a lemon-yellow to pale-yellow mineral, and occurs as an earthy powder, incrustations, fibrous masses, or capillary crystals in radiating groups. The crystals and many of the fibrous forms of the mineral have a silky luster. Molybdite is an alteration product of molybdenite, probably formed by the interaction of molybdic acid (liberated by the oxidation of the molybdenite) and limonite. The formation of the limonite in many cases may have resulted from the weathering of pyrite, which is commonly associated with the molybdenite, simultaneously with or previous to the weathering of the molybdenite.

Plate V, *B*, illustrates a group of molybdite crystals, showing their capillary form and characteristic grouping in radiating clusters. Plate II, *B*, shows a flake from a large crystal of molybdenite, with molybdite extensively developed along its cleavage planes.

For many years the erroneous idea has existed that molybdite agrees in composition with the artificial molybdic trioxide obtained by oxidizing molybdenite. The present textbooks on mineralogy almost universally persist in this error, giving the composition of molybdite as MoO_3, and stating that the pure mineral contains 66.7 per cent of molybdenum. That such is not the case was first called to the author's attention by chemical tests of a sample of molybdite which from microscopic examination seemed to be practically pure. The presence of a large percentage of iron was determined, and the same result obtained from tests on other samples of molybdite from different localities led the author to believe that the iron was an essential part of the mineral. A search of the literature on molybdite revealed the fact that Schaller[a] had not only proved the presence of combined iron in molybdite, but had definitely established its composition as $Fe_2O_3.3MoO_3.7\frac{1}{2}H_2O$.

His analyses of molybdite from different localities in the United States, given in the accompanying table, have the insoluble matter deducted and are recalculated to 100 per cent. The last column of the table gives the theoretical analysis as calculated from the formula $Fe_2O_3 3MoO_3.7\frac{1}{2}H_2O$. Attention is called to the close agreement of the analysis of the molybdite from New Hampshire with this calculated analysis.

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[a] Schaller, W. T., Mineralogical notes—series 1: U. S. Geol. Survey Bull. 490, 1911, pp. 84–92.

Analyses of molybdite.

Constituent.	Telluride, Colo.	Hortense, Colo.	California.	New Hampshire.	Calculated from formula.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
H_2O	16.8	20.19	19.5	18.28	18.57
Fe_2O_3	20.2	20.30	20.0	21.87	22.01
MoO_3	63.0	59.42	60.5	59.85	59.42
	100.00	100.00	100.0	100.00	100.00

The specific gravity of the molybdite from Hortense, Colo., is given by Schaller as 2.99.

TESTS FOR MOLYBDITE.

Regarding tests of molybdite, Schaller[a] writes as follows:

> On heating the mineral in a closed tube abundant water is easily given off and the mineral assumes a dark-olive color; on further heating it again becomes lighter in color. On heating the mineral in a crucible the color changes are very marked. At first the yellow mineral darkens and becomes a dark gray, appearing almost black and with slight olive tint, then it becomes a light yellow again, and on further heating changes to a deep orange color. If the mineral now be allowed to cool, the orange changes to yellow and back to orange again on reheating. If the dark-colored material be allowed to cool, it retains its dark gray color, and on reheating passes through yellow to the orange. On heating for some time at a higher temperature, the mineral, on cooling, becomes a permanent bright green. By further heating all of the molybdenum is volatilized and the dark-red ferric oxide remains. The mineral is readily soluble in hydrochloric acid, and is decomposed by ammonia, taking on a brown color (probably due to the separating ferric hydroxide). After a while, all the molybdenum of the mineral goes into solution, leaving the insoluble ferric hydroxide.

OCCURRENCE AND ACCOMPANYING MINERALS.

As molybdite is an alteration product of molybdenite, its occurrences and associated minerals are practically the same as those of the latter mineral. (See p. 11.)

THE RARE MOLYBDENUM MINERALS.

ILSEMANNITE.

Ilsemannite[b] is an oxide of molybdenum said to result from the decomposition of metallic molybdates and is probably identical with the artificial compound $MoO_2.4MoO_3$. It is a blue-black to black cryptocrystalline mineral, and has been reported as occurring in barite and associated with wulfenite at Bleiberg, in Corinthia. It is soluble in water, giving a deep blue solution which on evaporation yields dark-blue crystals. Lindgren and Ransome[a] state that material from a quartz vein carrying molybdenite, pyrite, and sphalerite and situated a short distance east of the Howard flat vein in the Cripple Creek District, Colo., upon exposure to the sun and air, turns yellow or green and finally a deep Prussian blue. Little tufts of yellow material are formed on the specimens and occasionally coatings of a dark-blue mamillary substance. "The yellowish material is believed to be molybdite." "The blue substance, which dissolves in water and is associated with some ferrous sulphate, also contains a large amount of molybdenum, and probably is the rare mineral ilsemannite ($MoO_2 4MoO_3$)."

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[a] Schaller, W. T., op. cit., pp. 85-86.

[b] Dana, E. S., A system of mineralogy, 6th ed., 1911, p. 202.

BELONESITE.

Belonesite[b] is regarded as magnesium molybdate ($MgMoO_4$). It is a white transparent mineral occurring in minute acicular crystals of the tetragonal system, and theoretically contains 52.08 per cent of molybdenum. It occurs in rock fragments enveloped in the Vesuvian lava of 1872.

TESTS.

Before the blowpipe belonesite fuses with difficulty. It dissolves readily in salt of phosphorus, but less easily in the borax bead, and is insoluble in acids.

POWELLITE.

Powellite is calcium molybdate ($CaMoO_4$), which when pure contains 47.98 per cent of molybdenum. It is a dull-gray color, and according to Schaller[c] has a specific gravity of approximately 4.25. It is a pseudomorph after molybdenite, often preserving the structure of the latter mineral and occurring in platy masses. Dana[d] states that calcium tungstate is present in the mineral and quotes an analysis showing 10.28 per cent WO_3. Schaller, however, calls attention to the fact that any calcium tungstate present exists as a mechanical mixture of scheelite with the mineral and states that "The association of molybdenite with scheelite is well known, and when the molybdenite alters to powellite the association of powellite with scheelite necessarily follows. The agencies affecting the change from molybdenum sulphide to calcium molybdate are apparently without effect on the scheelite."

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[a] Lindgren, Waldemar, and Ransome, F. L., Geology and gold deposits of the Cripple Creek district, Colo.: U S. Geol. Survey, Prof. Paper No. 54, 1906, p. 124.

[b] Dana, E. S., op. cit., p. 992.

[c] Schaller, W. T., Mineralogical notes—series 1: U. S. Geol. Survey, Bull. 490, 1911, pp. 80-83.

[d] Dana, E. S., op. cit., p. 989.

TESTS.

The mineral fuses at about 5 to a gray mass. It reacts for molybdenum with salt of phosphorus and is decomposed by nitric and hydrochloric acids.[a]

OCCURRENCE.

Powellite has been found in the United States in the Peacock lode of the Seven Devils mining district in western Idaho, where it is associated with argentiferous bornite and dark-brown garnet;[a] at the South Hecla copper mine, Houghton County, Mich., where it occurs in fine crystals;[b] near Oak Springs, Nye County, Nev.;[c] and Baringer Hill, Llano County, Tex.[d]

PATERAITE.

Pateraite[e] is supposed to be cobalt molybdate ($CoMoO_4$). It occurs as an impure massive mineral of black color associated with uranium in the Elias mine, Joachimisthal, Austria.

ACHREMATITE.

Achrematite[f] is a massive, crypto-crystalline mineral with the formula $3(3Pb_3As_2O_8PbCl_2)4(Pb_2MoO_5)$ and contains 3.40 per cent of molybdenum. It has an uneven to subconchoidal fracture, is brittle, has a hardness of 3 to 4, and a specific gravity of about 6. In color it is sulphur-yellow to orange and red, but in mass is liver-brown, owing to admixed limonite. Its streak is pale cinnamon-brown; its luster is resinous to adamantine and fragments of the mineral are translucent on thin edges. It occurs at the mines of Guanacere, Chihuahua, Mexico.

TESTS.

Before the blowpipe it decrepitates slightly, turning a dark brick red and fusing easily to a nearly black globule which shows indistinct crystalline facets on cooling. On charcoal it yields arsenical odors, a lead coating, and finally globules of lead

EOSITE.

Eosite[g] is supposed to be a vanado-molybdate of lead. Its exact composition is not considered as definitely established, although Gin[h] gives its formula as $Pb_3V_2MoO_{16}$. It occurs in minute square octahedral crystals of the tetragonal system which are found implanted on pyromorphite and cerussite at Leadhills, Scotland. It has a hardness of 3 to 4, and a brownish, orange-yellow streak. In color it is deep aurora-red.

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[a] Dana, E. S., A system of minerology, 6th ed., 1911, p. 989.

[b] Dana, E. S., op. cit., appendix I, p 55.

[c] Schaller, W. T., Mineralogical notes—series 1: U. S. Geol. Survey, Bull. 490, 1911, pp. 80–83.

[d] Schaller, W. T., loc. cit.

[e] Dana, E. S., op. cit., pp. 991–992.

[f] Dana, E. S., op. cit., p. 992.

[g] Dana, E. S., loc. cit.

[h] Gin, G., A memoir on the methods of treatment of simple and complex ores of molybdenum, tungsten, uranium, and vanadium: Trans. Am. Elec. Chem. Soc., vol. 12, 1907, p. 412.

TESTS.[a]

When heated in a closed tube, eosite darkens, but regains its color on cooling. When fused with potassium bisulphate it gives a mass that is slightly yellow while hot, but on cooling turns first reddish-brown, and finally a brownish orange-yellow. This mass, dissolved in water and boiled with metallic tin, colors the solution a faint greenish-blue. Eosite is not so rapidly attacked by hydrochloric acid as wulfenite or crocoite. When a splinter of the mineral is placed on a glass plate and treated with hydrochloric acid, with the subsequent addition of alcohol, and then gently evaporated, a blue to bluish-green coating is formed with a green precipitate on the edges.

A NEW MOLYBDENUM MINERAL.

F. B. Laney, of the United States Geological Survey, recently discovered a dark-green mineral rich in molybdenum, which occurs as an incrustation on specimens of ore from Vein No. 21 of the Lucania Tunnel, near Idaho Springs, Colo. Dr. Laney's examination of the occurrence, made in conjunction with the author, showed that the mine water, which percolated slowly from the vein in the immediate neighborhood of the green incrustations, contained a molybdenum compound that colored it a deep blue. This color was so intense that in several experiments in which the mine water was used as a writing fluid the writing was as legible as with an ordinary ink. The acidity of the water was pronounced, and in some places where it had dripped upon the iron mine rails it had eaten them to a depth of three-quarters of an inch or more.

An analysis of a sample of this water by R. C. Wells, of the United States Geological Survey, showed the following results:

Analysis of mine water from Vein 21, Lucania Tunnel, Idaho Springs, Colo.[b]

Substance.	Grams per liter.	Substance.	Grams per liter.
Molybdenum trioxide (MoO_3)	7.98	Magnesium	0.73
Molybdenum dioxide (MoO_2)	Trace.	Sodium	.26
Ferrous iron	2.01	Potassium	.14
Ferric iron	1.75	Sulphate (SO_4)	18.26
Aluminum	.27	Chlorine (Cl)	.17
Calcium	.50	Hydrogen (of free acid)	.13

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[a] Dana, E. S., loc. cit.

[b] The specific gravity of this water at 25° C. was 1.031.

There is no doubt that the color of the water is due to a dissolved molybdenum blue, possibly ilsemannite, but just what the compound is has not been determined.

The blue mine water had impregnated considerable areas of the vein material in the drift on both sides of the main tunnel and immediately adjoining it. The rock was colored a light blue with the coloring particularly strong along the fractures in the vein material and in the more porous rock. Where the rock carrying the molybdenum blue had been exposed to the air circulating along the main tunnel and about the mouths of the drifts there was a powdery incrustation of the dark-green molybdenum mineral already referred to. Several specimens were collected where this incrustation was a yellowish or golden green and a few where it was almost a bright yellow. On the dump at the mouth of the main tunnel many specimens were coated with the green molybdenum mineral. The latter had seemingly been formed by the action of the air on ore that had been impregnated with the molybdenum blue.

Owing to the difficulty of obtaining reasonably pure samples of these blue and green minerals, and to the inherent difficulties in the analysis of compounds, whose state of oxidation is so unstable, their exact composition has not as yet been determined.

The molybdenum minerals described above are probably identical with those noted by Lindgren and Ransome [a] in a small vein east of the Howard flat vein in the Cripple Creek district, Colorado. However, the author believes that the green molybdenum mineral (relatively insoluble) is formed from the soluble blue compound, and not vice versa, as indicated by Lindgren and Ransome.

DOUBTFUL SPECIES.

As already stated, there are other molybdenum minerals that have been reported besides those described above. Definite information concerning them is, however, so meager that their existence as distinct species is problematical. Principal among these doubtful minerals are molybdurane, molybdoferrite and knightite. The first two are referred to by Moissan [b] and also by Gin [c] who give the formula of molybdurane as $UO_2.UO_3.2MoO_4$, and of molybdoferrite as $FeMoO_4$. Des Cloizeaux [d] speaks of molybdurane as a molybdate of uranium, and says that it is found at Joachimsthal, Bohemia. Knightite, said to be a phosphate of molybdenum, is referred to by

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[a] Lindgren, Waldemar, and Ransome, F. L., Geology and gold deposits of the Cripple Creek district Colorado: U. S. Geol. Survey Prof. Paper 54, 1906, p. 124; also see pp. 12-13 of this report.

[b] Moissan, Henri, Traité de chimie minérale, t. 4, 1904, p. 685.

[c] Gin, Gustave, A memoir on the methods of treatment of simple and complex ores of molybdenum, tungsten, uranium, and vanadium: Trans. Am. Electrochem. Soc., vol. 12, 1907, p. 412.

[d] Des Cloizeaux, Alfred, Manuel de mineralogie, 1862, p. 271.

Hills,[a] who states that it was discovered by O. W. Knight, of Bangor, Me., in a molybdenite deposit at Catherine's Hill, of the same State.

Beside the foregoing minerals of molybdenum the presence of molybdenum in scheelite should be called to the reader's attention. Dana [b] by 17 analyses of scheelite from various parts of the world shows that molybdenum is present in this mineral in quantities varying from a trace up to about 8½ per cent.

PHYSICAL PROPERTIES OF MOLYBDENUM.

Pure molybdenum is a white metal. Its appearance depends largely on the method of production. If obtained by reducing the oxides or the sulphides of molybdenum by hydrogen, it is a gray powder which under heat and pressure may be compacted into a metallic bar that is brittle and even fragile. Produced by alumino-thermic methods or by reduction in the electric furnace, it is a compact metal, but owing to the absorption of carbon in the electric furnace, it is not pure and has different physical properties from the carbon-free metal, as is explained later. Pure compact molybdenum is malleable and is sufficiently soft to be filed and polished with ease. It will not scratch glass.

Within the last decade molybdenum was considered as absolutely lacking in ductility, but the research laboratory of the General Electric Co., at Schnectady, N. Y., has discovered a method of producing ductile forms of the element, which may be drawn into ribbons and fine wire.

Debray,[c] one of the earlier experimenters with the metal, considered it infusible, but although he failed to melt it, he discovered that at a high temperature it absorbed carbon. Moissan [d] subsequently obtained molten molybdenum in an electric furnace. Its exact melting point is still a matter of some question. The Bureau of Standards [e] has placed it about 2,500° C., or 4,500° F. This is over 1,400° C. above the melting point of copper, about 1,000° C. higher than that of iron, and 745° above that of platinum. Osmium, tantalum, and tungsten are the only three metals listed by the Bureau of Standards as having higher melting points. A resumé of melting-point determinations of molybdenum is given by Pirani and Meyer.[f]

Moissan [g] determined the specific gravity of molybdenum as 9.01,[h] but recent research has shown that this value is low, and further that the specific gravity of the metal increases appreciably with the amount of mechanical working to which it is subjected, a phenomenon common to many other metals, such as copper, zinc, and tungsten.[a] The specific gravity of ductile molybdenum before drawing was determined in the research laboratory of the General Electric Co. as 10.02, and after drawing it ranged from 10.04 for a wire 3.75 mm. in diameter to 10.32 for a wire 0.038 mm. in diameter.

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[a] Hills, B. W., The molybdenite deposits of Tunk Pond, Me.: Mining World, vol. 31, 1909, p. 323.

[b] Dana, E. S., A system of mineralogy, 6th ed., 1911, p. 987.

[c] Moissan, Henri, Traité de chimie minérale, t. 4, 1904, p. 687.

[d] Moissan, Henri, loc. cit.

[e] Stratton, S. W., Melting points of chemical elements: Bureau of Standards Circular 35, 1912, 2d ed., p. 2.

[f] Pirani, M. V., and Meyer, A. R., Über den Schmelzpunkt des Wolframs und des Molybdäns: Ver. deut. Phys. Gesell., Jahrg. 14, 1912, pp. 426–428.

[g] Moissan, Henri, loc. cit.

[h] Moissan, Henri, loc. cit.

The accompanying table [b] shows the tensile strength of molybdenum wire of various diameters in comparison with that of tungsten wire of the same sizes and with that of one size of hard-drawn piano wire. It will be noted that the values for molybdenum are approximately one-half those for tungsten and for the steel wire of corresponding size; likewise, that the tensile strength of both molybdenum and tungsten increases very appreciably with the fineness of the wire. In other words, the more the metals are worked the stronger they become.

Comparative tensile strength of molybdenum, tungsten, and steel wire.

Wire.	Diameter in thousandths of an inch.	Diameter in mm.	Pounds per square inch.	Kilograms per square mm.
Molybdenum	5.0	0.125	200,000 to 260,000	140 to 182
	2.8	.070	230,000 to 270,000	161 to 189
	1.5	.038	270,000 to 310,000	189 to 217
Tungsten	5.0	.125	460,000 to 490,000	322 to 343
	2.8	.070	480,000 to 530,000	336 to 371
	1.5	.038	550,000 to 600,000	385 to 420
Hard-drawn piano wire	3.0	.075	507,000	356

The electrical resistance of ductile molybdenum is 5.6 microhms per cubic centimeter for hard-drawn wire and 4.8 for annealed wire, the resistivity being measured at 25° C.[c] The temperature coefficient of electrical resistance between 0° and 170° C. is 0.0050.[d]

The specific heat of molybdenum as determined by Defacqz and Guichard[e] is 0.072 at 93° C., 0.074 at 281° C., and 0.072 at 440° C.

The emissivity of molybdenum measured with red light having a wave length 0.650 μ has been determined by Burgess and Waltenberg [f] at temperatures of 2,000° and 2,500° C. as 0.43 and 0.40, by Coblentz [g] at room temperature as 0.51, and by Mendenhall and

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[a] Fink, C. G., Ductile tungsten and molybdenum: Trans. Am. Electrochem. Soc., vol. 17, 1910, pp. 229-233.

[b] From Fink, C. G., loc. cit.

[c] Fink, C. G., loc. cit.

[d] Fink, C. G., loc. cit.

[e] Defacqz, E., and Guichard, M., Sur la détermination de la chaleur spécifique du tungstène et du molybdène: Anal. chim. Phys., ser. 7, t. 24, 1901, p. 139.

[f] Burgess, G. K., and Waltenberg, R. G., The emissivity of metals and oxides: Bureau of Standards Scientific Paper 242, 1914-15, p. 597.

[g] Coblentz, W. W., The reflecting power of various metals: Bureau of Standards Scientific Paper 152, 1911, p. 197.

Forsythe[a] at 1,000° and 2,400° C. as 0.44 and 0.37. Hasselberg[b] has determined the numerous lines of the spectrum of molybdenum.

As already stated, molybdenum produced by the reduction of molybdic oxide with carbon in an electric furnace does not possess the same physical properties as pure molybdenum, owing to its absorption of carbon. Metal obtained by this method is gray and brittle. It is also very hard and scratches steel and quartz; even the hardest file will not cut it when it contains a certain proportion of carbon. The melting point of the gray metal is much below that of pure molybdenum, and its specific gravity is also lower, ranging from 8.6 to 8.9, depending on the amount of carbon present. When pure molybdenum is surrounded with carbon and heated to about 1,500° C., it absorbs carbon and becomes hard. Inversely, if carbon-bearing molybdenum is melted with molybdenum dioxide, the carbon in the metal is oxidized and the molybdenum is refined and takes on the physical properties of the pure metal.[c]

CHEMICAL PROPERTIES OF MOLYBDENUM.

Metallic molybdenum is only slowly oxidized at ordinary temperatures, and drawn molybdenum wire retains its luster almost indefinitely. On prolonged heating at a dull-red heat it becomes covered with a white coating of molybdic trioxide, and at 600° C. it oxidizes rapidly and the trioxide formed sublimes.

Fluorine attacks molybdenum at ordinary temperatures, chlorine at a dull-red heat, and bromine at a cherry red, and iodine does not attack it appreciably at temperatures as high as 700° to 800° C. Molybdenum is readily attacked by nitric acid but is not affected by hydrochloric acid, and sulphuric acid attacks it only when hot and concentrated. Fused alkalies act only slowly on the metal, but fused oxidizing salts, such as potassium nitrate, sodium peroxide, and potassium chlorate, attack it rapidly.

Molybdenum combines with oxygen to form several oxides, the three most stable of which are the sesquioxide (Mo_2O_3), the dioxide (MoO_2), and the trioxide (MoO_3). The first two of these are basic, and few of their salts have been studied. The trioxide is acid. It is yellow when hot and white when cold. It is precipitated from solutions in hydrated form by nitric and hydrochloric acids, but is soluble in dilute sulphuric acid and the alkalies. When the hydrated oxide is gently ignited, it becomes dehydrated and much more difficultly soluble in dilute acids. When the trioxide is heated, it sublimes, sublimation commencing at 400° to 450° C.

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[a] Mendenhall, C. E., and Forsythe, W. E., The relation between blackbody and true temperatures for tungsten, tantalum, molybdenum, and carbon, and the temperature variation of their reflective power: Astrophys. Jour., vol. 37, 1913, pp. 380-390.

[b] Hasselberg, B., Die Spectra der Metalle im electrischen Flammenbogen; Spectrum des Molybdans: K. Svenska vet. Acad. Handb., Ny Föl., Bd. 36, No. 2, 1902, p. 1.

[c] Moissan, Henri, Traité de chimie minérale, t. 4, 1904, p. 689.

A large number of elements combine with molybdic oxide to form molybdates. Of these, sodium molybdate, Na_2MoO_4, potassium molybdate, K_2MoO_4, and ammonium molybdate, $(NH_4)_2MoO_4$, are the chief soluble salts of molybdenum. The first two of these are formed by fusion of the trioxide with the requisite amount of sodium potassium carbonate. The latter is formed by the action of concentrated ammonia on the trioxide. The heavy metals, such as barium, calcium, and lead, form molybdates which are insoluble, and use of this fact is made in analytical work. Besides the normal molybdates of the form X'_2MoO_4 many complex molybdates such as phospho-molybdates exist.

With the halogens molybdenum forms several compounds which vary greatly in stability. One compound, MoO_32HCl, is of importance as it is volatile at 250° C. and its formation affords a means of separating molybdenum from tungsten.

Molybdenum forms two principal sulphides, MoS_2 and MoS_3, both insoluble in dilute sulphuric and hydrochloric acids. These sulphides dissolve in the alkalies to form thiomolybdates of the order X'_2MoS_4, which react with acids to form sulphur, hydrogen sulphide, molybdenum disulphide, and molybdenum trisulphide.

Molybdenum combines with nitrogen, phosphorus, boron, carbon, and silicon. More complete references on the chemical properties of molybdenum are given by Roscoe and Schorlemmer,[a] who have been quoted freely in the foregoing discussion. Moissan [b] also gives detailed data on the chemistry of molybdenum.

USES OF MOLYBDENUM.

The principal use of molybdenum is in the manufacture of alloy steels to which, particularly in conjunction with chromium, manganese, nickel, cobalt, tungsten, and vanadium, it imparts many desirable properties. These steels are used for a large variety of purposes such as for crank-shaft and propeller-shaft forgings, high-pressure boiler plate, guns of large bore, rifle barrels, armor plate, armor-piercing projectiles, permanent magnets, wire, and for self-hardening and high-speed machine tools. Metallic molybdenum is used in various electrical contact making and breaking devices in X-ray tubes, and in voltage rectifiers, and in the form of wire for filament supports in incandescent electric lamps, and for winding electric resistance furnaces, and in dentistry. Molybdenum is also employed in the manufacture of chemical reagents, dyes, glazes, disinfectants, etc.

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[a] Roscoe, H. E., and Schorlemmer, C., A treatise on chemistry, vol. 2, 1913, pp. 1059–1082.

[b] Moissan, Henri, Traité de chimie minérale, t. 4, 1904, pp. 685–758.

MOLYBDENUM STEELS.

The early experimenters with the use of molybdenum in steel were almost unanimous in condemning the metal. Thomas Blair, of Sheffield, England, in a pamphlet published in 1894,[a] quotes a case "where 1 per cent of molybdenum rendered good iron red-short, and utterly worthless." The poor results obtained by most of the pioneer investigators were due to two main causes—first, the molybdenum powder or ferromolybdenum used in making the steels was generally highly impure, containing sufficient amounts of sulphur, oxides, and other deleterious substances to spoil any steel. Second, the few molybdenum steels that were prepared in such a manner as to be free from objectionable impurities were generally ruined by improper heat treatment. It is, therefore, not surprising that at the outset the element received a "black eye," and this bad reputation has been an important factor in delaying a more general recognition of the value of molybdenum in the manufacture of special steels.

Thomas Swinden was probably the first to conduct a thorough investigation of a series of strictly comparable carbon-molybdenum steels ranging in composition up to 8 per cent molybdenum and 1.2 per cent carbon. Besides proving the extreme susceptibility of these steels to heat treatment and that they were easily ruined by even a short heating at 950° C. followed by a prolonged cooling, he showed that the molybdenum increased the tendency of the steel to harden on cold working, and that the effects of oil quenching, followed by tempering, were increased by its presence. Further, his investigation demonstrated that the effect produced by molybdenum depended to a large degree upon the treatment of the steels. With normalized steels, molybdenum considerably increased the tensile strength with only a slight reduction of ductility, and this influence was most marked in high-carbon steels. Hardened and tempered steels containing 1 to 2 per cent of molybdenum showed extremely high tenacity values, accompanied by high ductility, but when steels with higher percentages of molybdenum were hardened and tempered, they actually became inferior. Annealed steels diminished in strength and ductility as the molybdenum content increased, and this diminution was noticeable when 0.9 per cent or more of carbon was present. Throughout Swinden's tests the influence of carbon in the presence of molybdenum was marked, and there is no doubt that, like tungsten, molybdenum assists in producing hardness by helping to retain the carbon in solid solution. The results of Swinden's work appear in two excellent papers.[b] It suffices here to say that in a general way molybdenum acts similarly to tungsten in steel, but that it is much more active—that is, less of it need be used to produce a given result, as might be suspected from its lower atomic weight (96) as compared with that of tungsten (184). It is impossible to give any absolute figures as to the relative influence of the two elements, as their effects are not exactly similar. Guillet [a] suggested that the effective ratio of molybdenum to tungsten was 1 to 4, but this is probably too high. Giesen [b] gives the ratio as 1 to 2.225, and from the tests correlated by Swinden it is seemingly between 1 to 2 and 1 to 3.

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[a] Tungsten and chromium alloys.

[b] Swinden, T., Carbon molybdenum steels: Carnegie Scholarship Memoirs, Iron and Steel Inst. (London) vol. 3, 1911, pp. 66-124; and A study of the constitution of carbon-molybdenum steels, ibid., vol. 5, 1913, pp. 100-168.

MANUFACTURE OF MOLYBDENUM STEELS.

The crucible process is generally used in the manufacture of molybdenum steel, and the molybdenum is added in the form of molybdenum powder, ferromolybdenum, or other molybdenum alloys. Molybdenum steel can, however, be made in the electric furnace, and also by the open-hearth process. It has also been produced on an experimental scale in the electric furnace directly from a mixture of hematite and molybdenite with coke, lime, and fluorspar.[c]

In adding molybdenum to steel it is generally advantageous to use ferromolybdenum, ordinarily containing about 80 per cent Mo, as the melting point of this alloy is several hundred degrees centigrade lower than that of the ordinary commercial brands of powdered molybdenum. Further, powdered molybdenum seemingly is more prone to oxidation than the ferro-alloy, but on the other hand it generally contains 4 or 5 per cent of various oxides of molybdenum which may aid in removing excess carbon.

As molybdenum is generally used in steel in conjunction with chromium, tungsten, nickel, vanadium, etc., it is often added in the form of alloys with these metals. Standard alloys of this type on the market are chrome-molybdenum, with 50 per cent molybdenum and 50 per cent chromium; molybdenum-nickel, with 75 per cent molybdenum and 25 per cent nickel; and ferromolybdenum-tungsten, containing molybdenum and tungsten in the proportion of 3 to 1. From 1 to 7 per cent vanadium, according to specifications, is sometimes added to the last-mentioned alloy.

USE OF MOLYBDENUM IN TOOL STEELS.

Molybdenum acts similarly to tungsten in the presence of chromium and manganese, and in combination with either of these elements and carbon it produces a self-hardening steel which is said to be a little tougher than the corresponding tungsten steel. A typical steel of this kind is one containing 4 to 6 per cent molybdenum, 1 to 2 per cent chromium, and 1.85 per cent carbon. Molybdenum likewise has the same properties as tungsten with reference to producing the high-speed qualities that enable a steel to retain its temper and hardness at a red heat. These qualities are developed by cooling the steel moderately fast from a high temperature, treatment that prevents the usual critical changes and keeps the steel in the austenitic condition. It is believed by many that high-speed steels produced with molybdenum are superior to the corresponding tungsten steels, both as regards toughness and durability. It is stated that they take a fine cutting edge. According to Gledhill,[a] one of the qualities of molybdenum high-speed steel is that it does not require such a high temperature in hardening as does tungsten steel, and he states that if a temperature of 1,000° C. is exceeded, the tools made from the steels are inferior and their life shortened. Carpenter[b] states, on the other hand, that molybdenum steels should be heated to 1,000° to 1,100° C. before they are cooled, whereas tungsten steel must be heated to about 1,200° C. The superior toughness of molybdenum high-speed steels is attributed to the fact that they contain more iron, as less molybdenum need be used to obtain the same result as with tungsten, and also because a lower heat is required in tempering them.

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[a] Guillet, Leon, Revue de Métallurgie, 1904, p. 390.

[b] Giesen, Walter, The special steels in theory and practice: Carnegie Scholarship Memoirs, Iron and Steel Inst. (London), vol. 1, 1909, p. 31.

[c] Keeney, R. M., The production of steels and ferroalloys directly from ore in the electric furnace: Carnegie Scholarship Memoirs, Iron and Steel Inst. (London), vol. 4, 1912, pp. 108-184. Describes production of molybdenum steel (pp. 173-175).

Certain difficulties, however, have been encountered in the use of molybdenum tool steels. Some users say that they are liable to crack in quenching, and others that they do not hold their cutting edge after retreatment as well as before. This deterioration in the steel upon repeated heating for dressing and treatment has been ascribed to the disappearance of molybdenum from the outer skin of the steel through volatilization. In a few instances service tests with these steels have shown irregular cutting speeds and have indicated a tendency of the molybdenum to render the tools brittle and weak in their bodies. Some users have found that molybdenum tool steel was apt to be seamy and to contain physical imperfections, also that it is apt to fire-crack during treatment. Molybdenum tool steels of high carbon content require great skill in their preparation owing to the difficulty of judging by color the definite temperature required for hardening. Further, great care in annealing molybdenum steel is necessary after it has been worked into bars and before it is cut into shapes for tools, and previous to hardening.

Some of the objectionable features mentioned above, and perhaps most of them, are due to the use of impure ingredients in the manufacture of the steels or to improper heat treatment, and undoubtedly many of them can be overcome. However, most manufacturers, particularly in this country, have almost entirely discontinued the use of molybdenum as a major constituent in tool steel and are now using it largely in a minor capacity in conjunction with tungsten, cobalt, etc. Used in this way many of the difficulties mentioned above have been overcome, and the resulting steels have at the same time required certain superior qualities imparted by the molybdenum. A characteristic steel of this type is one with 16 to 18 per cent tungsten, 1.5 to 2 per cent molybdenum, 4 to 4.5 per cent chromium, and 0.6 per cent carbon, and it is said to be superior in cutting efficiency to the corresponding tungsten-chromium steel. It is also stated that the addition of the molybdenum in this steel results in a finer texture and lessens the chances of injuring the steel previous to hardening. The addition of molybdenum is particularly common in high-speed steels where cobalt is used in conjunction with tungsten. This type of steel contains about 16 to 18 per cent tungsten, 4 to 5.5 per cent cobalt, and 0.25 to 1.5 per cent molybdenum. Small percentages of vanadium are sometimes used in connection with molybdenum and tungsten in tool steels and are said to increase their strength and cutting efficiency. An analysis of such a steel containing both vanadium and cobalt in conjunction with tungsten, chromium, and molybdenum, and known as iridium steel, follows:

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[a] Gledhill, J. M., The development and use of high-speed tool steel: Jour. Iron and Steel Inst., No. 11, 1904, pt. 2, pp. 127-182.

[b] Carpenter, High-speed tool steels: Jour. Iron and Steel Inst., No. 1, 1905, p. 460.

Approximate average analysis of iridium steel.[a]

Constituent.	Per cent.	Constituent.	Per cent.
Cobalt	4.25	Manganese	Low.
Tungsten	16.00	Silicon	
Chromium	3.55	Sulphur	
Vanadium	.67	Phosphorus	
Molybdenum	.80	Carbon	0.60
		Iron	[b] 74.00

It is stated that this iridium steel will do 60 per cent more work than the best tungsten steel.

The introduction of high-speed tool steels has completely revolutionized machine-shop practice. Gledhill[c] states that, as compared with cutting speeds of 30 to 50 feet per minute for ordinary crucible steel, high-speed steels are now made that will cut 500 feet per minute on steel, and drill cast iron at the rate of 25 inches per minute. Planing speeds of 15 to 25 feet per minute have been increased to 50 and 60 and even to 80 feet per minute. The high-speed twist drill has also made radical changes in practice. To-day it is customary to stack small plates and drill them instead of punching them, not only saving time, but avoiding the liability of injuring the metal by straining it. The plates for torpedo boats are now drilled instead of punched. It was formerly the custom to core in the holes in pipe flanges, but they are now drilled.

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[a] Hess, F. L., Cobalt, Mineral Resources, U. S., 1912, U. S. Geol. Survey, 1913, p. 967.

[b] Approximate.

[c] Gledhill, J. M., The development and use of high-speed tool steel: Jour. Iron and Steel Inst., No. 11, 1904, pp. 127-182.

USE OF MOLYBDENUM IN MAGNET STEEL.

The molybdenum steels used in making permanent magnets are similar in composition to the high-speed molybdenum steels. They generally contain 2 to 3 per cent molybdenum, 0.5 to 0.7 per cent carbon, and sometimes about 0.5 per cent chromium. After hardening they retain their magnetism longer than hardened carbon steel, and are said to be superior to tungsten magnet steels. Some steels of this type contain up to 6 per cent molybdenum.

USE OF MOLYBDENUM IN ACID-RESISTING STEELS.

Alloyed with chromium, and sometimes with chromium and tungsten, molybdenum is used in preparing acid-resisting steels. Borchers [a] states that an alloy containing 2 to 5 per cent molybdenum, about 10 per cent chromium, and a little or no carbon, is practically acid proof. Molybdenum-tungsten-chromium-iron alloys have also been made that are insoluble in hydrochloric, sulphuric, or nitric acid, and an alloy containing 60 per cent chromium, 35 per cent iron, and 2 to 3 per cent molybdenum is said to resist even the action of boiling aqua regia.[b] Differences in the heat treatment of these alloys have a great effect on their acid-resisting qualities.

USE OF MOLYBDENUM IN OTHER ALLOY STEELS.

Molybdenum is used in conjunction with chromium and nickel to produce steels with a wider heat-treatment range and with even greater tensile strength than are possessed by chrome-nickel steel. These chrome-nickel-molybdenum steels contain about 1 per cent chromium, 2 to 3 per cent nickel, and 0.25 to 0.5 per cent molybdenum, and on account of their high tensile strength and elastic limit, they have a range of usefulness that is limited only by their cost. These properties make them of special value for crank shafts, propeller shafts, and other machine parts that are subjected to alternating and repeated stresses. For example, they are used to some extent in the frames and axles of automobiles and railroad rolling stock. The high tensile strength of these steels is also responsible for their use in the best grades of high-pressure boiler plate such as is used on torpedo boats. They are also employed in making guns of large bore, and rifle barrels, and for such use they have the added advantage of being highly resistant to the erosive action of the gases generated by the explosives.

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[a] Borchers, W., Acid-resisting alloys; Engineer (London), vol. 114, July 26, 1912, p. 83.

[b] Benner, R. C., Recent advances in industrial chemistry: Min. and Sci. Press, vol. 105, 1912, p. 629.

It has been found that the addition of a small percentage of molybdenum is advantageous in the manufacture of armor plate and shields for small guns. The molybdenum is said to make the steel softer when it is annealed so that it can be planed more readily, and upon hardening, the molybdenum content produces an increased tensile strength. The exact compositions of the various molybdenum steels used in armor plate are, of course, carefully guarded secrets. Stoughton[a] states that Krupp armor plate (probably of early manufacture) contains about 3.25 per cent nickel, 1.5 per cent chromium, and 0.25 per cent carbon. The assumption is safe that the introduction of 0.25 to 0.5 per cent of molybdenum in a steel of this composition would increase both its hardness and its tensile strength.

The use of molybdenum in connection with chromium is reported for producing steels of great hardness and high elastic limit, which are employed in the manufacture of armor-piercing projectiles. About 1 per cent of molybdenum added to a nickel steel is said to impart to it remarkable drawing qualities.

FERROMOLYBDENUM.

Ferromolybdenum has been used for electrodes in arc lamps.[b]

USE OF MOLYBDENUM IN STELLITE.

Small percentages of molybdenum are used in certain patented nonferrous alloys consisting essentially of chromium and cobalt. These alloys are known under the trade name of "Stellite," and possess remarkable high-speed qualities when used for machine tools. They are also employed for cold chisels, wood-working tools, cutlery, etc. Their use in cutlery is of particular interest as they do not tarnish under atmospheric influences and are unaffected by fruit acids.

In so far as molybdenum is concerned, two stellite alloys are of interest, namely, those containing cobalt, chromium, and molybdenum, and those containing cobalt, chromium, tungsten, and molybdenum. Haynes,[c] the inventor of these alloys, says:

> When molybdenum is added to a 15 per cent cobalt-chromium alloy [15 per cent chromium], the alloy rapidly hardens as the molybdenum content increases, until the content of the latter metal reaches 40 per cent, when the alloy becomes exceedingly hard and brittle. It cuts keenly and deeply into glass, and scratches quartz crystal with ease. It takes a beautiful polish, which it retains under all conditions, and on account of its extreme hardness its surface is not readily scratched. When 25 per cent of molybdenum is added to a 15 per cent chromium alloy, a fine-grained metal results, which scratches glass somewhat readily, and takes a strong, keen edge. Its color and luster are magnificent, and it will doubtless find a wide application for fine, hard cutlery. It can not be forged, but casts readily, and its melting point is not abnormally high.

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[a] Stoughton, Bradley, The metallurgy of iron and steel, 1908, p. 407.

[b] Editorial, Molybdenite: Min. and Sci. Press, vol. 104, 1912, p. 781.

[c] Haynes, Elwood, Alloys of cobalt with chromium and other metals: Trans. Am. Inst. Min. Eng., vol. 44, 1912, pp. 576-577.

These stellite alloys are silvery-white. They are insoluble in nitric acid and are only slowly attacked by hydrochloric acid. Following is the analysis of a typical molybdenum stellite.

Analysis of a typical molybdenum stellite.[a]

Constituent.	Per cent.
Mo	22.50
Co	59.50
Cr	10.77
Fe	3.11
Mn	2.04
C	.87
Si	.77
S	.084
P	.040
W	.00
Ni	.00
	99.684

USES OF METALLIC MOLYBDENUM.

The uses of metallic molybdenum have been greatly extended by the discovery of certain processes by which it can be obtained in ductile form.[b] As wire, it is used for supporting the filaments in incandescent electric lamps. Of course, the quantity of the element used in a single lamp of ordinary candlepower is extremely small, but in argon-nitrogen-tungsten lamps of high candlepower the molybdenum supports are of very appreciable size and weight. Molybdenum wire is also employed for winding electric resistance furnaces, and for this use it has proved both cheaper and superior to platinum because of the quicker heating and the higher temperatures attainable.[c] In this work it must, of course, be protected from the air to prevent its oxidation.

Molybdenum has been successfully substituted for platinum and for platinum iridium in various electric contact making and breaking devices. On account of its high heat conductivity in the ductile form, and because its relative cheapness permits its use in large masses, the formation of heavy coatings of nonconducting oxide are prevented, and under the conditions existing in these contacts, any thin coatings of oxide formed are conductors. The Coolidge X-ray tube and a voltage rectifier of recent manufacture have a very considerable proportion of ductile molybdenum used in their construction. Gold-covered molybdenum wire is used in dentistry to a considerable extent, and research work is being carried on to extend the use of special molybdenum alloys in this field.[d]

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[a] Hibbard, H. D., Manufacture and uses of alloy steels: Bull. 100, Bureau of Mines, 1915, p. 61.

[b] See Coolidge, W. D., U. S. Patent 1082933, Dec. 30, 1913.

[c] Winne, R., and Dantsizen, C., Small electric furnace with heating element of ductile tungsten or ductile molybdenum: Jour, Ind. and Eng. Chem., vol. 3, October, 1911, p. 770.

[d] Fahrenwald, F. A., A development of practical substitutes for platinum and its alloys, with special reference to alloys of tungsten and molybdenum: Am. Inst. Min. Eng., Bull. 109, January, 1916, pp. 103-149.

It is suggested that molybdenum may be successfully used with tungsten as a thermocouple for the measurement of high temperatures. The advantage in the use of these metals is that they have much higher fusing points than platinum, platinum-iridium, or platinum-rhodium couples, such as are ordinarily used, and although they oxidize at high temperatures, they might be protected from the air by covering them with fused magnesia or similar substances. The voltage curve of the molybdenum-tungsten couple has been studied by Northrup.[a]

USE OF MOLYBDENUM IN CHEMICALS.

The chief use of molybdenum in chemicals is as ammonium molybdate, $(NH_4)_2MoO_4$, and sodium molybdate, $Na_2\ MoO_4$. The first is employed principally as a reagent in the quantitative determination of phosphorus in iron and steel, various ores, fertilizers, soils, etc., and in the quantitative determination of lead. The equivalent of several tons of metallic molybdenum is said to be consumed annually in this form by the iron and steel plants in the United States for the determination of the phosphorus content of their products. It is reported that ammonium molybdate is also used for fireproofing, and as a disinfectant for cloth used in railroad coaches and for similar purposes. It is said to be a strong germicide. Sodium molybdate was at one time used rather extensively in ceramics to impart a blue color to pottery and in the manufacture of certain glazes, but it is understood to have been replaced almost wholly in this field by cobalt compounds. It is also used in dyeing silks and woolens. Molybdenum indigo, Mo_5O_7, is used for coloring rubber and is said to be noninjurious to the material.[b]

Fast colors in a large variety of shades may be produced on leather by employing molybdenum tannate in conjunction with logwood extracts.[c]

A process involving the use of molybdenum for the preservation of cordite in hot climates is said to have been discovered in France.[d] It is also rumored that molybdenum is used in the synthetic production of ammonia under processes covered by German patents and in which it is said to act as a "promoter" to the catalyzers used. It is well known that ammonia may be made by heating molybdenum oxide or molybdenum hydroxide, or mixtures of the two, to about 500° to 600° C. with equal equal parts of nitrogen and hydrogen, and under a pressure of about 60 atmospheres, to form molybdenum nitride, which when heated in a vacuum yields ammonia and metallic molybdenum.[a]

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[a] Northrup, E. F., Tungsten and molybdenum; their thermal E. M. F.: Met. and Chem. Eng., vol. 11, January, 1913, p. 45.

[b] Hess, F. L., Tungsten, nickel, cobalt, etc.: Mineral Resources U. S. for 1908, U. S. Geol. Survey, 1909, p. 746.

[c] Pozzi-Escot, E., Production de coluers fixes sur tous genres de cuirs, par l'emploi de sels de molybdène combinés à des matierès tannantes ou à des coluers mordantes vegetales: Compt. rend., t. 135, 1902, p. 801.

[d] Editorial, Molybdenite: Min. and Sci. Press, vol. 108, 1914, p. 860.

PRODUCTION OF MOLYBDENUM ORES.

Queensland, New South Wales, and Norway have to date furnished the major part of the world's production of molybdenum, the output derived from all other countries, including the United States, probably not amounting to 10 per cent of the total. Austria, Canada, France, Germany, Great Britain, Japan, Mexico, Natal, Russia, Sweden, the United States, and Australian States other than those mentioned above have at times made a small production of molybdenum ore, but with the exception of the United States their output has been too small to be worthy of consideration.

The following table shows the tonnage and value of high-grade molybdenite concentrates produced in Queensland, New South Wales, and Norway from 1902 to 1914:

Production of high-grade molybdenite concentrates in Queensland, New South Wales, and Norway, 1902 to 1914.

Year.	Queensland.[a]		New South Wales.[b]		Norway.[c]	
	Short tons.	Dollars.	Short tons.	Dollars.	Short tons.	Dollars.
1902	[d] 45.9	[d] 26,770	16.8	8,960	22	16,100
1903	[d] 26.9	[d] 10,220	32.5	21,690	34	21,400
1904	23.6	13,010	28.3	13,270	33	17,400
1905	70.8	41,340	21.7	12,200	51	16,300
1906	118.9	74,330	36.6	23,350	[e] 1,129	14,200
1907	74.0	41,080	24.2	17,340	33	12,900
1908	98.7	44,960	9.5	4,520	39	13,400
1909	103.9	45,120	31.5	15,810	33	12,100
1910	118.6	58,640	53.2	27,580	....	....
1911	111.4	64,610	23.1	12,610	2	800
1912	114.6	84,420	63.3	18,030	23	5,400
1913	74.3	92,460	88.3	33,100	13	3,200
1914	87.1	185,830	68.8	55,720	....	....

[a] Data from annual reports of the under secretary of mines, Queensland.
[b] Data on quantity exported from annual report, Department of Mines, New South Wales, 1914.
[c] Data from Norges Bergvaerkdrift, Norges Officielle Statistik.
[d] The molybdenite production of Queensland in 1902 and 1903 as officially reported includes a small tonnage of bismuth and tungsten ore.
[e] Probably ore.

QUEENSLAND.

The first official record of a production of molybdenite in Queensland was in 1900, when the output amounted to 12.3 short tons of high-grade material. The production gradually increased to 118.9 short tons in 1906, and it varied little from this figure until 1913, when it dropped to 74.3 tons, but rose again in 1914 to 87.1 tons. The value of the production in the latter year, however, showed an enormous increase, owing to an advance in prices, and amounted to $185,830, or more than twice the value of the 1913 production, which was only slightly less in tonnage. The total production to the end of 1914 was approximately 1,030 short tons, valued at about $760,000. The bulk of this material was mined at Wolfram Camp, in the Chillagoe field, about 120 miles southwest from Cairns, in northern Queensland. The mines at Bamford, in the same field, are, however, credited with a small production. In 1914 the principal producing properties in the Wolfram Camp district were the Murphy & Geaney mine, which treated 1,233 long tons of ore, which yielded 20.2 tons of molybdenite, valued at £9,543, and 83.4 tons of bismuth-tungsten concentrates, valued at £5,558, and the Larkin & Wade mine, which made a total output of 1,254 long tons of milling ore, which yielded 9.65 tons of molybdenite and 54.55 tons of bismuth-tungsten concentrates. Other mines at Wolfram Camp which made smaller productions of molybdenite were the Gillian Lease, Tulley, Tulley Block, Hillside United, Smith United, and Mulligan & McIntyre. At Bamford the Trafalgar, Evening Star, Northern United, Morning Star, and Sunny Corner mines made a total output of 8.55 long tons of high-grade molybdenite.

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[a] Tucker, S. A., and Moody, H. R., The production of hitherto unknown metallic borides: Jour. Chem. Soc., vol. 81, 1902, p. 16.

The total production of the Chillagoe district in 1914 was 76.25 long tons of molybdenite. The remainder of the output came from the Star River (Ollera) district and from a newly discovered field at Glen Atherton, on Upper Tinaroo Creek, in the Gladstone district.

Detailed descriptions regarding the occurrence and production of molybdenite in Queensland are given in the annual reports of the acting undersecretary for mines and in the references following:

Literature on occurrence and production of molybdenite.

Saint-Smith, E. C., Molybdenite in the Stanthorpe-Ballandean districts, Southern Queensland: Queens. Govt. Min. Jour., vol. 15, 1914, pp. 184–189.

Cameron, W. E., Wolfram, molybdenite, and bismuth mining at Wolfram Camp, Hodgkinson goldfield: Queens. Govt. Min. Jour., vol. 4, 1903, pp. 350–352.

Cameron, W. E., Wolfram and molybdenite mining: Queens. Govt. Min. Jour., vol. 5, 1904, pp. 62–65.

Ball, L. C., The wolfram, molybdenite, and bismuth mines of Bamford: Geol. Surv. Pub. 248, Mines Dept., Queensland.

Ball, L. C., Rare-metal mining in Queensland—résumé of recent field studies: Queens. Govt. Min. Jour., vol. 14, pp. 4–7.

NEW SOUTH WALES.

The production of molybdenite in New South Wales was first reported in 1902. In that year the output was 16.8 short tons, valued at $8,960. The total production to the end of 1914 was 498 short tons, valued at over $264,000.

The principal producing molybdenite mines are at Whipstick in the Pambula division, at Kingsgate in the Glenn Innes division, and near Deepwater in the Deepwater division. The annual report of the Department of Mines for the year 1914 states that at Whipstick the Whipstick Mines (Ltd.) raised 63 long tons of ore, estimated to contain 9 tons of concentrates, valued at £5,000. At Kingsgate, in the Glenn Innes division, the Yates mine produced 8.75 tons of concentrates, containing 94 per cent molybdenite and valued at £4,700, and the Glenn Innes Molybdenite and Bismuth Syndicate sold 1.6 long tons of concentrates containing 92 per cent molybdenite for £655. The Sacks mine at Kingsgate produced 0.85 ton of molybdenite, valued at £340. In the Deepwater division the Bow Creek Molybdenite Mines (Ltd.) produced 65 long tons of ore, which yielded 1.63 tons of concentrates, valued at £735. In the same division 11 tons of ore, which yielded 1.5 tons of molybdenite concentrates, valued at £700, was mined by E. A. Baker, and 28 tons of ore, which yielded 1.4 tons of molybdenite, assaying 93 per cent MoS_2, and valued at £667, was mined at Four Mile by E. L. Key. At Rocky River, in the Tantafield division, 80 tons of molybdenite ore, valued at £631, was mined, and in the Bathhurst division, 38 tons, valued at £800. The table on page 29 shows that the greatest production of molybdenite in New South Wales was in 1913, when 88 short tons, valued at $33,100, was mined, and that in 1914 the output decreased to 68.8 tons. On account of the marked advance in prices, however, the value of the output in the latter year was more than $20,000 in excess of the value of the production in 1913.

Detailed descriptions of the occurrence and production of molybdenite in New South Wales are given in the annual reports of the department of mines, New South Wales, and by Andrews.[a]

NORWAY.

In Norway the production of high-grade molybdenite concentrates has averaged about 30 tons per annum since 1902. The table on on page 29, in which the output of this country is given by years, shows that in 1906 a production of 1,129 short tons was reported. This figure doubtless does not represent the output of a high-grade molybdenite concentrate, but rather the production of ore in that year.

The principal molybdenite-producing sections in Norway are the Provinces of Lister og Mandal and Nedenes, on the extreme southern end of the peninsula. The district of Fjotland, in the former Province, is probably richer in molybdenite than any yet discovered in Norway. A mine at Knaben, in this district, has been the largest and probably the only successful producer in Norway. This mine, owned by George G. Blackwell & Sons Co., of Liverpool, England, has made an average output of about 25 short tons per annum. In 1912 about 200 tons of low-grade molybdenite ore was mined at Reinshommen, in Fjotland, and about 610 tons of ore averaging about 2.4 per cent MoS_2 at Langvand. Of this latter amount 390.5 tons was milled, yielding 0.5 metric ton of concentrates, assaying 51 per cent of MoS_2, and 10 tons of second concentrates running about 23 per cent MoS_2.[a]

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[a] Andrews, E. C., Molybdenum: New South Wales Dept. of Mines and Agriculture, Geol. Survey Min. Resources, No. 11, 1906, 17 p.

It is reported that on account of the increased demand and high prices being paid for molybdenum a considerable interest is being taken at the present time in developing some of the numerous Norwegian deposits. The Ore Concentration Co. (Ltd.), of London, has recently tested several parcels of Norwegian molybdenite ore aggregating over 100 tons. This company states that the Elmore oil-flotation process is in use at two mines in Norway, and that large quantities of molybdenite concentrates have already been shipped.[b]

UNITED STATES.

The principal production of molybdenum ore in the United States was in 1903, when, according to Pratt,[c] 795 short tons of wulfenite and molybdenite concentrates, valued at $60,865, was marketed. Probably 750 tons, or more, of this material consisted of wulfenite concentrates obtained by sluicing tailings from a cyanide mill at Mammoth, Ariz. The ore from which these tailings were derived came from the Mammoth mine at Schultz, about 3 miles away. The remainder of the output, consisting of molybdenite concentrates, was probably derived from the Crown Point mine, Chelan County, Wash., with perhaps a ton or two from the mine of the American Molybdenum Co., at Cooper, Me., and from other sources. Previous to 1903 practically the entire commercial output of molybdenum ore in this country consisted of 20 to 30 tons of high-grade molybdenite obtained from the Crown Point mine.[d] From 1903 to 1914 the United States Geological Survey has recorded a production of a marketable molybdenum product in only three years, namely, 1905, 1906, and 1907. The output in these three years was confined to small lots of molybdenite derived largely from the Crown Point mine and from a deposit near Homestake, Mont., and to a few tons of wulfenite concentrates from Arizona.[a] Official statistics as to the production in 1914 are as yet not available, but it is known to have been small and to have consisted for the most part of wulfenite concentrates obtained from a small mill erected by F. H. Hereford and R. O. Boykin, of Tucson, Ariz., to re-treat the tailings piles already mentioned at Mammoth, Ariz. Small lots of high-grade molybdenite coming from the Crown Point mine, Chelan County, Wash., and the Romero Mining Co.'s property near Porvenir, N. Mex., were also marketed in 1914.

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[a] Editorial, Norwegian molybdenite: Eng. and Min. Jour., vol. 98, 1914, p. 820.

[b] Editorial, Molybdenum recovery by the Elmore process: Eng. and Min. Jour., vol. 99, 1915, p. 907.

[c] Pratt, J. H., The steel-hardening metals: Mineral Resources U. S. for 1903, U. S. Geol. Survey, 1904, p. 308.

[d] Pratt, J. H., Tungsten, molybdenum, uranium, and vanadium: Mineral Resources U. S. for 1901, p. 268, and for 1902, p. 287, U. S. Geol. Survey, 1902 and 1903.

Since January, 1915, the production of both molybdenite and wulfenite in this country has shown a notable increase. In October, 1914, the Primos Chemical Co., of Primos, Pa., purchased a molybdenite prospect on Red Mountain, near Empire, Colo., and developed the property to such an extent that shipments of ore were made in January, 1915, and it is understood that they continued at the rate of two to three 25-ton carloads per week until the mine was shut down for the winter. It is reported that the ore shipped contained 2 to 3 per cent of molybdenite. Since the beginning of 1915 an assaying and ore-testing firm of Denver, Colo., has purchased considerable quantities of molybdenite ore coming from various mines in this country and Canada, and is said to have marketed a small tonnage of molybdenite concentrates of good grade derived from these ores. The embargo placed by Great Britain on the exportation of molybdenum ore from her colonies has of course since prevented further receipt of Canadian ores by this company. It is reported that a contract was placed with another company of Denver, Colo., in 1915, for the concentration of 500 tons of low-grade molybdenite ore from a deposit on Bartlett Mountain, Summit County, Colo., and, further, that several hundred tons of low-grade ore from neighboring claims were concentrated by flotation with fair results at Leadville, Colo. Considerable development work was done on these deposits during the year. Noteworthy development, resulting in the production of a considerable tonnage of low-grade ore, was likewise done by the Leviathan Mines Co. on its molybdenite property near Copperville, Mohave County, Ariz. Several molybdenite prospects throughout the West produced small quantities of ore, and small lots of high-grade molybdenite were obtained from some of them by careful selective mining on streaks of high-grade material and by hand picking.

The production of wulfenite also increased. F. H. Hereford and R. O. Boykin enlarged their mill at Mammoth, Ariz., to over twice its previous capacity, and during the summer were producing a wulfenite concentrate containing approximately 22 per cent MoO_3 at the rate of about 2 tons per day.

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[a] Pratt, J. H., Steel and iron hardening metals: Mineral Resources U. S. for 1905, U. S. Geol. Survey, 1906, p. 412. Hess, F. L., Nickel, cobalt, vanadium, etc.: Mineral Resources U. S. for 1906, U. S. Geol. Survey, 1907, p. 539. Hess, F. L., Tungsten, nickel, cobalt, etc.: Mineral Resources U. S. for 1907, U. S. Geol. Survey, 1908, p. 721.

A small production of wulfenite concentrates were said to have been derived in 1915 by Mr. Frank Widener, of Cutter, N. Mex., from a deposit about 30 miles west of that place. Considerable development work was done on the Old Yuma mine near Tucson, Ariz., and a small mill was erected for the treatment of the wulfenite ore by Col. Epes Randolph and associates of Tucson.

The author believes that the output of molybdenum contained in the molybdenite and wulfenite ores mined in the United States in 1915 will compare favorably with that of any other country. Whether it will continue to increase in the future depends entirely on the demand.

CANADA.

The production of molybdenum ore in Canada has to date been confined to small lots of high-grade molybdenite obtained by cobbing and hand-picking ore from rich streaks in certain deposits in the Provinces of Ontario and British Columbia. It is reported that these lots, which have usually averaged 500 to 2,000 pounds in weight, have been largely sold to chemical manufacturers in the United States, although a few of them have been shipped to England. In addition to this output of high-grade material perhaps 100 to 200 tons of medium-grade molybdenite ore has been shipped to the United States or to England for concentration. Of course, since the embargo placed by Great Britain on the exportation of molybdenum ore from her colonies such shipments to the United States have ceased. Although up to the present time no production worthy of mention has been made in Canada, that country has several promising deposits of high-grade molybdenite ore and is doubtless capable of a considerable production.

IMPORTS OF MOLYBDENUM.

Under the tariff of 1909 the duty on molybdenum or ferromolybdenum valued at $200 per ton or less was 25 per cent ad valorem, and on material valued at more than $200 per ton it was 20 per cent ad valorem. Under the existing tariff this duty was reduced to 15 per cent ad valorem, irrespective of value. Ores of molybdenum may be imported free. Although it is known that small lots of high-grade molybdenite have been brought into this country at frequent intervals, no figures as to the quantity so imported can be given, as no separate account of the imports of molybdenum ore are kept by the customs service. However, the total quantity is probably not large, as practically the entire output of Queensland, New

South Wales, and Norway, which have been the principal producing countries to date, has been taken by Germany, England, and France. It is understood that during the early part of 1915 about 60 tons of high-grade molybdenite ore was imported from Canada for concentration in this country; but the action of the British Government in placing molybdenum ores, metallic molybdenum, and ferromolybdenum on the list of absolute contraband has made impossible further shipments of such material into this country, either from Canada or other British possessions.

The table following shows the amount and value of the molybdenum and ferromolybdenum imported into the United States during the years 1911 to 1915. Previous to 1911 imports of these metals were not separately reported by the customs authorities. It will be noted that during the period covered by the table imports of these metals were practically negligible.

Quantity and value of imports of molybdenum and ferromolybdenum into the United States, 1911 to 1915.

Calendar year.	Tons.	Dollars.
1911	8.5	11,409
1912	4.0	4,670
1913	7.0	15,939
1914	.1	59
1915	2.0	2,370

MARKET FOR MOLYBDENUM.

Under normal conditions practically 90 per cent of the world's molybdenum production is used in making alloy steel and only about 10 per cent in the manufacture of chemicals and for all other purposes. At first glance it is therefore somewhat surprising that with the greatly increased demand for alloy steels occasioned by the European war the market for the element should not have developed to a larger degree, and in a greater measure kept pace with the phenomenal demand for its sister metal, tungsten, especially as molybdenum has in the past been used principally in the manufacture of field artillery, coast-defense and naval guns, rifle barrels, armor plate, armor-piercing projectiles, etc., and of high-speed tool steel, which is finding such a large use in the manufacture of these and other munitions. The ultimate market for molybdenum has, however, always been largely in Europe, the use of the element in steel being much more popular abroad than in the United States. Up to the present time France, England, and Germany have taken practically the entire molybdenum output of the world, and in comparison an almost negligible quantity of molybdenite and wulfenite, derived from the small domestic production and small quantities of molybdenite imported, have been treated in this country. Outside of a few companies that are purchasing high-grade molybdenite concentrates for the manufacture of chemicals, there are even to-day only five concerns in the United States that are making metallic molybdenum or ferromolybdenum. Of course, there are many ore buyers in this country who are in the market for molybdenite concentrates, but their market is in turn almost wholly abroad, and they act either as middlemen, on their own behalf, or as agents for European purchasers.

The development of the molybdenum market has in the past and is at present being retarded by the fact that manufacturers who might use or might investigate the possibilities of using the metal are kept out of the market by the fear of not being able to obtain steady supplies. On the other hand, those who might be interested in the development of some of the extensive low-grade molybdenite or wulfenite deposits in this country are prevented from doing so by the small visible demand and the fear that any large production will glut the market. Owing to these conditions, the mining of molybdenum has, as already stated, been confined in the past to small-scale operations on high-grade streaks of molybdenite ore, and the methods of recovery have been limited largely to cobbing and hand picking. The unprecedented demand for steel-hardening metals, occasioned by the European war, is, however, slowly drawing the attention of both prospective producers and consumers to the possibilities in regard to molybdenum. Several mining operations looking toward a considerable production of both molybdenite and wulfenite have already been commenced, and, on the other hand, manufacturers are investigating the possibilities of using molybdenum in a large way, and are beginning to realize that it is not as they generally supposed, due to the scarcity of the element in nature and the nonexistence of ore bodies that steady supplies have previously not been available, but rather to lack of development of these ore bodies.

How great a quantity of molybdenum the market will absorb under existing or future conditions is extirely a matter of speculation. It is the author's opinion, however, that the demand for the element will keep pace with a greatly increased production, particularly if the metal is offered at prices below those normally asked for tungsten.

HOW MOLYBDENUM CONCENTRATES ARE MARKETED.

The basis on which molybdenum ores and concentrates are bought and sold varies according to whether the contained molybdenum mineral is molybdenite or wulfenite. Molybdenite products are invariably purchased on the basis of their molybdenum content, reckoned as MoS_2, whereas wulfenite is always bought either on the basis of its molybdenum content expressed as metallic molybdenum or as MoO_3. It is unfortunate that custom should have established these three methods of calculation where one, based on the content of metallic molybdenum, would have sufficed and avoided needless complications and frequent misunderstandings between buyers and sellers. In connection with the use of these three standards it may be of interest to note that 1 part by weight of MoS_2 is equivalent to 0.9 part of MoO_3 and to 0.6 part of Mo, and, inversely, that 1 part by weight of Mo is equivalent to 1.5 parts of MoO_3 and to 1.67 parts of MoS_2.

In the United States the short ton of 2,000 pounds is the measure of weight used in buying and selling molybdenum ore, and quotations are generally based on the number of units of 20 pounds each of pure Mo, MoO_3, or MoS_2 contained in a ton. In Europe, on the other hand, the long ton of 2,240 pounds is almost invariably used, and accordingly European purchasers settle on the basis of a unit containing 22.4 pounds. Quotations both in this country and abroad are generally made on a sliding scale to cover various grades of material. Specifications usually state the minimum percentage of Mo, MoS_2, or MoO_3 in the ore or concentrates that is acceptable to the purchaser, and also the maximum percentage of objectionable elements, such as copper, tungsten, bismuth, arsenic, and antimony that will be allowed. Just what are objectionable elements depends largely on the use to which the molybdenum product derived from the concentrates is destined and the methods employed in treating it. Copper and tungsten seem to be universally objectionable both to the iron and steel and to the chemical trades. Copper is particularly undesirable and its presence in excess of 1 per cent, even in high-grade molybdenite concentrates, is usually sufficient to render the material unmarketable. Some dealers will, however, accept concentrates containing more than 1 per cent copper, but when 4 or 5 per cent copper is present the penalties exacted are such as to be prohibitive. Likewise, some dealers have no objection to the presence of small quantities of arsenic and bismuth in molybdenite concentrates, as they state that these elements are readily volatilized when the ore is roasted in the process of treatment. Up to 1914 it was difficult to sell molybdenite concentrates containing less than 80 per cent MoS_2, or wulfenite concentrates with less than 25 per cent MoO_3, but at present (March, 1916) it is reported that molybdenite and wulfenite concentrates containing as low as 20 per cent MoS_2 and 18 per cent MoO_3, respectively, can be marketed.

PRICES OF MOLYBDENITE AND OF WULFENITE CONCENTRATES.

Under the existing abnormal conditions of the market for all steel-hardening metals it is impossible to quote exact prices for either molybdenite or wulfenite concentrates. The sale of even 1-ton or 2-ton lots is the subject of separate negotiations, and the prices received vary between wide limits. Based on a content of 90 per cent MoS_2, small lots of molybdenite have, during the last two years, brought in this country $2,500 to $3,500 per short ton, or $27.75 to $38.90 per 20-pound unit of MoS_2. The price of wulfenite concentrates during the same period has ranged from $200 to $300 per short ton, or from $10 to $15 per unit of MoO_3, based on a content of 20 per cent MoO_3. Of course, the price paid per unit for MoS_2 or MoO_3 decreases rapidly with the grade of the material, as is well illustrated by the following schedule of prices quoted in February, 1916, by a domestic manufacturer of molybdenum and ferromolybdenum, for molybdenite concentrates. Based on a content of 50 per cent MoS_2, the price offered was $20 per unit of MoS_2, with a penalty or bonus of 20 cents per unit for each per cent below or in excess of 50 per cent MoS_2. The prices of various grades of concentrates, according to this schedule, are shown in the accompanying table:

Prices offered for molybdenite concentrates in February, 1916.

Per cent MoS_2 in concentrates.	Price offered—	
	Per unit.	Per short ton.
30	$16	$480
40	18	720
50	20	1,000
60	22	1,320
70	24	1,680
80	26	2,080
90	28	2,520

As already stated in the discussion of the market for molybdenum, concentrates of both molybdenite and wulfenite must be practically free from deleterious elements, such as copper, tungsten, antimony, arsenic, and bismuth, to command the best prices, and a copper content of more than 2 or 3 per cent generally precludes their sale or else exacts heavy penalties. The following table, which was compiled mainly from quotations furnished through the courtesy of L. Lamy, of Paris, France, shows the fluctuations in the price of high-grade molybdenum concentrates (90 to 95 per cent MoS_2) in European markets since 1907. The table shows that the price has more than trebled in the past three years:

Prices of high-grade molybdenite concentrates (90 to 95 per cent MoS_2) in European markets, 1908 to 1915.

Year.	Prices per unit of 20 pounds.	Prices per unit of 22.4 pounds.	Year.	Prices per unit of 20 pounds.	Prices per unit of 22.4 pounds.
		Shillings.			*Shillings.*
1908	$6.50 to $7.60	30 to 35	1912	$7.15 to $11.95	33 to 55
1909	5.65 to 7.15	26 to 33	1913	10.90 to 18.50	50 to 85
1910	6.30 to 6.75	29 to 31	1914	19.50 to 31.50	90 to 145
1911	6.95 to 9.10	32 to 42	1915	23.90 to 36.90	110 to 170

In conclusion, it may be said that prices at present are wholly abnormal, and companies engaged in the mining or concentration of either molybdenite or wulfenite should be prepared for the sharp decline that is almost certain to follow the cessation of hostilities in Europe. Just what the normal price will be is hard to predict, as there are too many uncertain factors to be considered. As already stated, it is the opinion of the author that the demand for molybdenum will continue to increase, particularly if the price of the metal declines so that it closely approaches the normal price of tungsten. Any large production of molybdenum, such as might be derived from the several extensive deposits of low-grade molybdenite ore that are now being developed in this country, would, in all probability, not only cause such a decline in price, but might possibly enable molybdemun to be sold even below the normal price of tungsten. Although it is not safe to make any prediction as to the minimum price at which molybdenum may be marketed in the future, it may be assumed that if the chief use of the metal continues to be in the manufacture of special steels, maximum prices will never greatly exceed twice the corresponding price of tungsten, as the effective ratio of the two metals in steel is slightly above 1:2, and in many instances steel manufacturers could substitute one for the other within certain limits without materially affecting their product.

The present abnormal prices for molybdenite and wulfenite concentrates are, of course, reflected in the quotations on metallic molybdenum and ferromolybdenum. Prices in this country (March, 1916) are nominal at $4 to $5 per pound of metallic molybdenum, whereas in England they are slightly lower, averaging about 15s. 6d. or $3.75 per pound. It is of interest to note that inasmuch as supplies of molybdenum and tungsten are of vital importance in the manufacture of munitions, Great Britain, acting through the governments of her various colonial possessions where ores of these metals are produced, has established a maximum price at which these ores may be sold. As regards molybdenum ore this price is 105s. per unit of MoS_2, and for tungsten ore it is 55s. per unit of WO_3. Taken in connection with the placing of these metals and their ores and alloys on the list of absolute contraband, this fixing of prices by the British Government has resulted in an extraordinary difference in the relative prices of the two metals in the United States and Great Britain. Quotations on ferrotungsten (75 to 85 per cent tungsten) are (March, 1916) about $8.50 per pound of contained tungsten in this country as compared with 6s. 6d. to 7s. ($1.58 to $1.70) in Great Britain. In other words, molybdenum is being sold in the United States at less than half the price of tungsten, whereas in Great Britain the reverse is the case. However, this great difference in price is not to be wholly accounted for by the conditions mentioned above, but it is partly due to the fact that the use of molybdenum is much more popular abroad than in this country. For example, some well-known European metallurgists are understood to prefer to purchase molybdenite ore at 120s. per unit of MoS_2 rather than tungsten ore at 55s. per unit of WO_3. In this country, on the other hand, the dêmand for tungsten is far in excess of that for the comparatively little-known molybdenum.

QUALITATIVE TESTS FOR MOLYBDENUM.

One of the best and most easily applied qualitative tests for molybdenum is made by treating the finely powdered ore with about 5 c. c. of concentrated nitric acid, evaporating to dryness, treating the residue with ½ c. c. of concentrated sulphuric acid and again evaporating to dryness. If molybdenum is present a beautiful ultramarine blue color develops in the residue on standing. This characteristic color generally appears in two hours or less, but sometimes 12 hours may be required, depending on the temperature and the humidity.

Another test that is highly recommended is to fuse the finely powdered ore with a mixture of sodium and potassium carbonates (1 to 1) and a small amount of potassium nitrate. The fusion is powdered and extracted with hot water and filtered, the filtrate acidified with hydrochloric acid, the carbon dioxide expelled by boiling, and potassium or ammonium thiocyanate, together with a small piece of metallic zinc, added. If molybdenum is present a bright cherry-red color rapidly develops which disappears on standing in the presence of the zinc. If hydrogen peroxide is added to the solution immediately after the cherry-red color has developed, the color disappears, returning as soon as the peroxide has been reduced. If only small proportions of molybdenum are present, it is suggested that immediately after the thiocyanate and zinc have been added to the acidified filtrate from the leached carbonate fusion, the solution be shaken with a small volume of ether. Any color in the liquid will be extracted by the ether and intensified as a golden brown in the ether ring that forms at the top of the liquid on standing, thus perhaps making visible color that before was imperceptible.

Should there be an imperfect fusion and any extraction of iron in the leach from the fused carbonates, a cherry-red color will immediately develop on the addition of the potassium or ammonium thiocyanate to the solution. With the addition of zinc, however, the color due to iron disappears, and unless a large quantity of iron has been brought into solution the color will completely disappear prior to the development of any color due to molybdenum. If this color reaction of iron and its disappearance on the addition of zinc is borne in mind, the above test for molybdenum is infallible.

Blowpipe and simple chemical tests for the various molybdenum minerals are given on pages 8 to 15.

QUANTITATIVE DETERMINATION OF MOLYBDENUM.

Previous to the quantitative determinations of the molybdenum in the ores and products obtained therefrom in the concentration tests described in this report, a brief study of published methods for determining the element was conducted by J. C. Morgan, junior chemist, in the Denver office of the Bureau of Mines, to whom the author is indebted for data in the following discussion.

It was found that most of the published methods were open to objections as hereinafter outlined. Further, several precipitation methods that would, without doubt, give correct results on practically pure salts of molybdenum were absolutely inaccurate when applied to certain ores and particularly to low-grade ores. In view of the difficulties encountered, a brief preliminary discussion of the more common methods for the quantitative determination of molybdenum may be of interest here.

SOLUTION.

The general methods of getting the molybdenum in an ore into solution are as follows: (1) By fusion with a mixture of sodium and potassium carbonates and subsequent leaching of the molybdate formed with hot water; (2) by fusion with a mixture of sodium carbonate and sulphur (1 to 1) and the leaching of the sulpho-molybdate formed with hot water; (3) by fusion with sodium peroxide or sodium hydroxide followed by leaching the molybdate formed with hot water;[a] (4) by solution obtained through evaporation, first with fuming nitric acid, and then with concentrated sulphuric acid.[b]

The fusion methods all have one common disadvantage for ores containing silica in excess of 10 per cent or thereabouts, in that the greater part of the silica is brought into solution and must be removed previous to the precipitation of the molybdenum. Otherwise the molybdenum precipitate is contaminated with silica. The removal of the greater part of the silica necessitates a tedious evaporation, with subsequent filtration and washing of the precipitate. Further, if the alkaline solution is evaporated as in Low's method,[c] the precipitated silica is gelatinous and difficult to wash free from molybdenum salts. If the solution is acidified with hydrochloric acid, as is usual in the precipitation of silica, molybdic trioxide is precipitated on evaporation. One advantage of the fusion methods is that lead, copper, bismuth, iron, and aluminum are left in the residue from the leach, thus precluding the necessity of their removal later.

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[a] Darroch, James, and Meiklejohn, C. A., A rapid method of determining molybdenum: Eng. and Min. Jour., vol. 82, 1906, p. 818.

[b] Colett, E., and Eckhardt, M., Bemerkungen zur Bestimmung des Molybdäns im Molybdänglanz: Chem. Ztg., Jahrg. 33, 1909, p. 968.

[c] Low, A. H., Technical methods of ore analysis, 1913, p. 185.

The method of solution by digesting with fuming nitric acid followed by evaporation to dryness, fuming with a small quantity of concentrated sulphuric acid, and diluting, brings all of the molybdenum into solution, if the final residue is washed with dilute ammonia. Any silica or lead present is left in the residue. In contrast to results with fusion methods, copper, bismuth, iron, and aluminum are brought into solution. The removal of these elements, however, seems to offer less difficulty than the removal of silica in the fusion methods.

PRECIPITATION.

Many methods have been advanced for the precipitation of molybdenum from solution. The chief of these are as follows: (1) Precipitation as molybdenum trisulphide, (2) precipitation as mercurous molybdate, (3) precipitation as lead molybdate, and (4) precipitation as barium molybdate.

The complete precipitation of molybdenum as molybdenum trisulphide may be accomplished in several ways, as follows: (1) By saturating a cold solution, slightly acid with hydrochloric or sulphuric acid, with hydrogen sulphide and heating in a pressure flask;[a] (2) by saturating an alkaline solution with hydrogen sulphide, acidifying with either hydrochloric or sulphuric acids, and heating in a pressure flask;[b] (3) by saturating an ammonical solution with hydrogen sulphide, acidifying with hydrochloric acid, coagulating, filtering, evaporating the filtrate to dryness, expelling the greater part of the ammonium salts, extracting the residue with water containing a small amount of ammonia, adding ammonium sulphide, and acidifying with hydrochloric acid, thus removing the last traces of molybdenum;[c] (4) by adding thioacetic acid and heating the precipitated molybdenum sulphide in a pressure flask.

The precipitation of mercurous, lead, or barium molybdate is accomplished by adding a solution of a salt of these metals to the molybdate solution. With sulphide ores the precipitation of either lead or barium molybdate is impracticable, as no matter what method of solution is used there is invariably enough sulphate present to cause a considerable error through the precipitation of lead or barium sulphate. The prevention of the precipitation of lead sulphate by the use of either sodium or ammonium acetate in hot solution when precipitating lead molybdate is not advisable, owing to the large quantity of acetate required.

The precipitation of mercurous molybdate is usually accomplished by adding a slightly acid mercurous nitrate solution to the solution obtained from the carbonate fusion, to which nitric acid has been added until the solution is only slightly alkaline.[a] Considerable trouble is experienced in obtaining the right degree of alkalinity. Care must be taken not to obtain an acid solution, as molybdic trioxide may precipitate.

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[a] Treadwell, F. P., Analytical chemistry, vol. 2, 1912, pp. 285-286.

[b] Treadwell, F. P., loc. cit.

[c] Colett, E., and Eckhardt, M., loc. cit.

CONVERSION OF PRECIPITATE TO WEIGHABLE FORM.

Molybdenum may be weighed as MoO_3, MoS_2, $PbMoO_4$, or $BaMoO_4$. When precipitated as MoS_3 there are two courses open—either ignition to MoO_3 or to MoS_2 in a stream of hydrogen, according to the Rose method. The fact that MoO_3 is readily volatile at temperatures higher than 400° to 450° C. necessitates great care in igniting. The complete conversion of the MoS_3 to MoO_3 requires long ignition at a temperature that should not greatly exceed 350° C. The regulation of this temperature by the ordinary Bunsen flame is extremely difficult and requires much practice. In reducing to MoS_2 many ignitions are sometimes required to obtain a constant weight.

The conversion of mercurous molybdate to molybdic trioxide by volatilizing the mercury requires extreme care and much time, and it is almost impossible to expel all the mercury without volatilizing some of the molybdic oxide. Lead and barium molybdate precipitates are weighed as such after the precipitates have been dried.

VOLUMETRIC DETERMINATION OF PRECIPITATED MOLYBDENUM TRISULPHIDE.

Several volumetric methods have been offered for the determination of molybdenum, but all require that the molybdenum first be freed from the other metals. These methods are, therefore, only of value when applied to ore analysis to determine the molybdenum from a re-solution of the final sulphide precipitate. A valuable volumetric method for such a determination is that of Randall.[b] In this method a solution of the sulphide, acidified with sulphuric acid, is passed through a zinc reductor into a ferric iron solution, and the ferrous iron formed is titrated with permanganate. The advantage that volumetric methods have over gravimetric methods is that they eliminate tedious and difficult ignitions.

In general, practically all molybdenum ores carry silica, together with such metals as iron, aluminum, copper, and lead, so that the most desirable method of analysis would be one in which these elements could be eliminated most expeditiously and completely and give a final molybdenum precipitate readily convertible to a weighable form. A well-tested method that does this most efficiently is that of Colett and Eckhardt.[c]

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[a] Low, A. H., loc. cit.

[b] Randall, D. L., The behavior of molybdic acid in the zinc reductor: Am. Jour. Sci., ser. 4, vol. 24, 1907, p. 313.

[c] Colett, E., and Eckhardt, M., loc. cit.

METHOD ADOPTED BY THE BUREAU OF MINES.

Inasmuch as the ores treated carried only traces of arsenic or antimony and no tungsten, no account was taken of these metals in the procedure employed. Special precautions are necessary when these metals are present in appreciable quantity. The procedure for the separation of molybdenum from these metals may be found in any of the more complete texts on molybdenum analyses.

Directions covering the method adopted by the Bureau of Mines follow:

Digest the sample of ore—from 0.2 gram to 5 grams, depending upon its seeming richness—with 25 to 35 c. c. of fuming nitric acid in an Erlenmeyer flask for three hours and finally evaporate to dryness. Add 3 c. c. of concentrated sulphuric acid to the residue and heat until dense white fumes are given off in quantity. Cool, dilute to 100 c. c., and filter. Wash the residue with water, allowing the wash water to run into the filtrate. Wash the residue well with dilute ammonia (1 to 3), and then with water. Make the filtrate alkaline with ammonia to precipitate the aluminum and any iron present in the original mineral. Heat, filter, and wash well with hot water. Saturate this alkaline filtrate with hydrogen sulphide to a bright cherry-red color. Filter and wash with hot water. Acidify the filtrate with hydrochloric acid until slightly acid and digest until the precipitated sulphide and sulphur are well coagulated and the excess hydrogen sulphide expelled. Filter on a weighed Gooch crucible. Evaporate the filtrate to dryness in a casserole and drive off the ammonium salts at the lowest possible temperature, being careful not to heat the casserole to redness at any time. Take up the final residue with about 100 c. c. of water to which 5 c. c. of ammonia has been added. Add 10 c. c. of ammonium sulphide, make faintly acid with hydrochloric acid, and digest until the sulphide is coagulated. Filter this on the Gooch crucible used for the previous sulphide filtration. Add an amount of sulphur to the combined sulphides equal to about one-half their weight and ignite over a Bunsen burner at a dull-red heat in a stream of arsenic-free hydrogen for 10 minutes. The ignition may be accomplished by using a Rose crucible cover and tube over the Gooch crucible. Weigh and repeat the ignition as before, until check weights are obtained. The weight obtained is the weight of molybdenum disulphide.

PART II. DESCRIPTION OF DEPOSITS.

GENERAL DISTRIBUTION OF DEPOSITS.

A brief field investigation confined to the examination of the better known occurrences of molybdenum in six of the Western and Pacific Coast States, namely, Arizona, California, Colorado, Montana, New Mexico, and Washington, has been sufficient to show that, contrary to general belief, there are in this country large low-grade deposits of both molybdenite and wulfenite ores from which considerable tonnages of marketable concentrates might be obtained.

Examinations of the various deposits were made solely with the purpose of determining their commercial possibilities. Most of those visited were in the prospect stage, and as many of those in the States of Colorado, Montana, and Washington were seen in the late fall when they were covered with snow, some of the following descriptions may seem to appear brief and incomplete. It is hoped, however, that the descriptions and the tabulated information regarding the known occurrences of molybdenum ores in the various States will be of value to those who are interested in deposits of this metal.

ARIZONA.

Arizona has many deposits of molybdenite and of wulfenite, and with the exception of Nevada it is the only State in which any noteworthy deposits of the latter mineral have been reported. Wulfenite ores from Arizona have supplied the larger part of the molybdenum produced in this country to 1915, and on account of the ease with which they are concentrated they should prove a strong competitor of molybdenite for markets that do not require concentrates with a high molybdenum content.

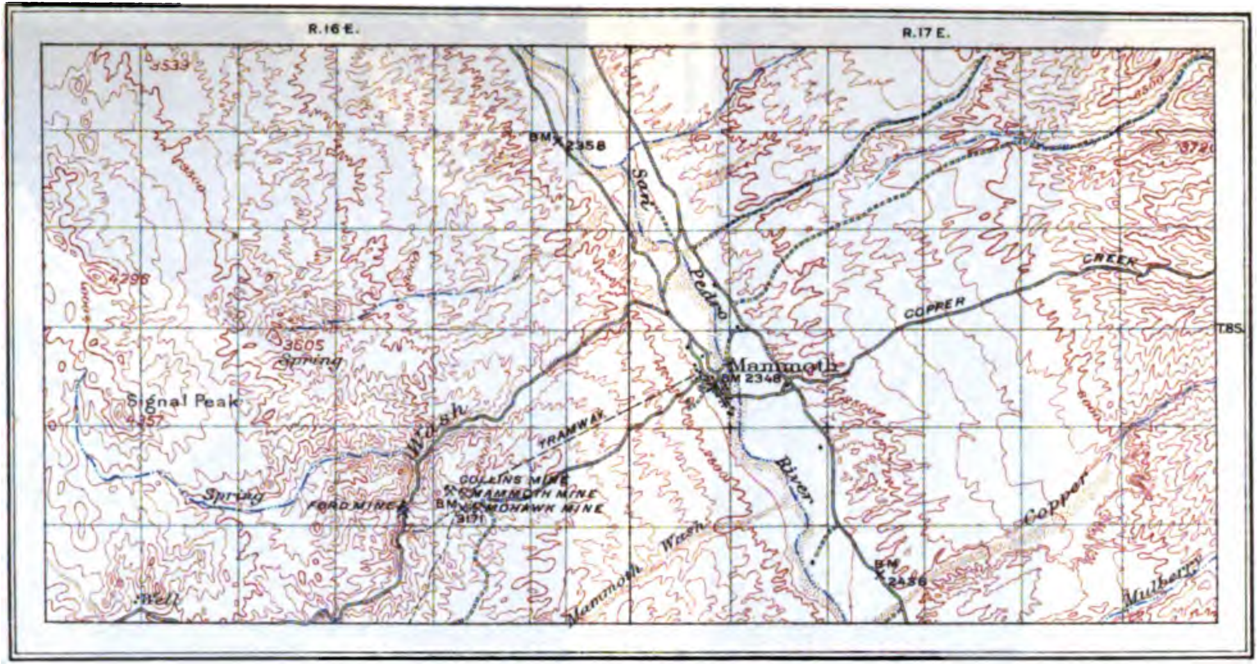
The deposits of wulfenite ore are largely confined to the four southern counties, Cochise, Pima, Pinal, and Yuma, with a few in Gila, Maricopa, and Yavapai Counties. Molybdenite has been reported from only six counties—Greenlee, Gila, Mohave, Pima, Pinal, and Santa Cruz. In most of the Arizona deposits the molybdenite is associated with chalcopyrite and other copper minerals, and inasmuch as the copper in the concentrates must be separated by further treatment the deposits are not as desirable as those free from copper. However, notwithstanding the unfortunate association of copper, some of these deposits will doubtless prove of commercial importance.

WULFENITE AT THE MAMMOTH AND COLLINS MINES, PINAL COUNTY.

The Mammoth and the Collins are adjoining mines situated at Schultz, in the Old Hat mining district, Pinal County, about 3 miles west of Mammoth and 48 miles north-northeast from Tucson, the railroad point from which supplies are brought over a good wagon road via Oracle. The mines are in section 26, T. 8 S., R. 16 E., in hilly, desert country, on the easterly slope of the drainage basin of the San Pedro River. Their location with reference to Mammoth and the main topographic features of the vicinity are shown in Plate VI. A view of the buildings and dump of the Mammoth mine is given in Plate VII, *A*. No wood for fuel or for mine timbers is available near by. Fuel oil and distillate for power and wood for timbering are hauled from Tucson. The elevation at the collar of the Mammoth shaft is 3,213 feet, and the collar of Collins shaft about 750 feet to the southwest is 65 feet higher.

The mines are situated on two roughly parallel veins that are about 600 to 700 feet apart. The veins strike in a general northwest to southeast direction and dip to the southwest. The dip of the Mammoth vein varies from 45° to 72°. Its average dip between the 300-foot and the 750-foot levels is 69° 31′, but at the 750-foot level it is only about 45°. From the surface to the 300-foot level the average dip of the Collins vein is about 72°, but grows flatter below. The veins vary greatly in width, both along the strike and the dip. The Mammoth vein is 1 or 2 inches to 60 feet wide, the part that has been explored south of the main shaft and that roughly constitutes "the south ore shoot" averaging 13 to 15 feet wide for a horizontal distance of about 750 feet. Work on the Collins vein indicates that it has an average width of 12 to 13 feet.

The filling of the Mammoth vein seems to be a mixture of brecciated rhyolite and granite, the whole so highly altered and silicified that its exact classification is impossible. The ore contains wulfenite, vanadinite, descloizite, cerussite, anglesite, chrysocolla, azurite, malachite and small quantities of gold and silver. A few pockets of galena carrying a little silver are found in some of the stopes. The filling of the vein in the Collins mine is similar to that of the Mammoth vein except that it consists almost entirely of altered rhyolite(?). The accompanying minerals in the two veins seem to be identical. The geologic history of the veins is complex and has not been thoroughly studied. Speaking in a general way, however, one may say that they were formed by intrusions of rhyolite(?) along faults in the granitic country rock, the whole being subsequently shattered by faulting and mineralized. The character of the rock of the walls is very irregular; in some places the hanging wall is rhyolite and the



MAP OF THE VICINITY OF MAMMOTH, ARIZONA,

SHOWING LOCATION OF MAMMOTH MINE

Scale $\frac{1}{125000}$ 1 ½ 0 1 2 3 4 5 Miles Contour interval 100 feet.

A. BUILDINGS AND DUMPS OF MAMMOTH AND COLLINS MINES, SCHULTZ, ARIZ.

B. CRYSTALLIZED WULFENITE (BLACK) IN LEAD CARBONATE (WHITE) FROM MAMMOTH MINE, SCHULTZ, ARIZ. (NATURAL SIZE).

foot wall granite, and vice versa. Seemingly the zone of mineralization repeatedly crossed and recrossed the shattered rhyolite. The primary deposition of the economic minerals was probably as sulphides, and the existing minerals may be considered as almost wholly of secondary origin.

Wulfenite is of common occurrence throughout the veins and much of it is beautifully crystallized. The Mammoth mine alone has probably furnished more fine cabinet spicimens of the crystallized mineral than any other locality. Most of the crystals are light orange, but they range through various shades of orange, brown, and green, to almost black. Plate III, *B*, shows a remarkably fine group of crystals from this mine. In many places there are 1-inch and 2-inch streaks of nearly pure wulfenite near the center of the ore bodies, and many cracks and fractures throughout the vein material are filled with the mineral. On the 750-foot level of the Mammoth mine the writer noted a shoot of wulfenite and cerussite ore, exposed for 100 feet or more, that averaged 10 to 12 inches wide, and probably contained 20 to 30 per cent of wulfenite. Plate VII, *B*, shows a characteristic occurrence of a veinlet of crystallized wulfenite in cerussite. In many cases the wulfenite is intimately associated with vanadinite, whereas in others the two minerals occur separately.

Veinlets and minute stringers of wulfenite are visible throughout the stopes and in the roofs and floors of most of the drifts. The writer estimates that as a whole the ore bodies contain perhaps 1 to 2 per cent of the mineral. The mines have, however, never been worked for wulfenite, but have been developed chiefly for gold, of which their ores are said to carry $6 to $7 per ton. Years ago, 225,000 to 250,000 tons of ore were taken from the Mammoth mine and treated by stamp milling and amalgamation at Mammoth, 3 miles away. Later the old tailing piles were treated for gold by the cyanide process, and still later parts of them were sluiced to extract the wulfenite. At present these tailings are being re-treated for the third time for their wulfenite content, as described on pages 113 to 115.

Several years ago control of both the Collins and the Mammoth mines, which had been idle for many years, was obtained by the Messrs. Young, who installed new machinery and other equipment and did considerable development work with a view of active mining.

Development on the Mammoth mine consists of a main shaft 833 feet deep and about a mile of drifts on eight different levels. The workings on the north side of the shaft have caved badly to within 125 feet of the shaft and are mostly inaccessible. On the 750-foot level it is, however, possible to go 350 feet north of the shaft, but only with great danger. The vein at this level on the north side varies from 6 to 60 feet wide. On the south side of the shaft the principal work has been done on the first, third, fourth, fifth, sixth, and seventh levels, and aggregates perhaps 4,000 feet of drifts. Much ore has been stoped, but the ground has not caved to any extent. The water level in the Mammoth mine is about 760 feet below the collar of the shaft.

The Collins mine is opened by a tunnel 550 feet long and by a shaft down 700 feet. The principal drifts are as follows: One 325 feet long on the 100-foot level, and one 300 feet long on the 200-foot level. In all, the Collins mine has perhaps 2,000 feet of tunnel and drifts.

The Mammoth mine is equipped with a shaft house, engine and compressor house, boiler house, machine and blacksmith shops, oil storage tanks, etc. Power is generated by two Fairbanks-Morse engines which use "tops" or a No. 2 distillate costing (June, 1915) about 5 cents per gallon in Tucson. One of these engines, rated at 100 horsepower, is belted to a Sullivan angle-compound compressor capable of delivering 528 cubic feet of air per minute at 100 pounds pressure. The other engine, rated at 150 horsepower, is direct connected to a 440-volt generator which supplies power for the mine pump, electric lights, and various small motors used in the machine shop, etc. The hoisting equipment consists of a 12-inch by 14-inch double-drum hoist, which can be operated either by steam or compressed air. The mine pump is situated on the 750-foot level. It is a Prescott direct-connected motor-driven pump with a lifting capacity of 400 gallons per minute against a head of 900 feet.

Both the Mammoth and the Collins mines could, without doubt, produce a large tonnage of low-grade wulfenite ore containing perhaps 2 to 3 per cent of the mineral, and with careful selective mining could produce considerable high-grade ore. It is the writer's opinion, however, that a high-grade wulfenite concentrate could not be produced by treating the ore as a whole by ordinary wet methods on account of its containing considerable cerussite. In fact, a concentrate obtained in this way from ore of ordinary grade from the Mammoth mine showed on analysis only 14.45 per cent molybdic trioxide (corresponding to 39.4 per cent of wulfenite), but 54.54 per cent lead. The vanadinite and copper minerals present in the ore would also tend to detract from the value of the product. However, concentrates of the grade indicated above are salable, and ways might be devised for separating the bulk of the cerussite. It is reported that the wulfenite from these mines contains a small amount of gold.

WULFENITE AT THE OLD YUMA MINE, PIMA COUNTY.

The Old Yuma mine is situated about 14 miles northwest of Tucson, and 5 miles southeast of the Southern Pacific Railroad in the foothills of the Tucson mountains. To reach it one follows an excellent county road for a distance of 11 miles and then takes a poor road for about 3 miles. The mine is in desert country, and neither water nor timber is available in the immediate neighborhood. At the mine an inclined shaft had been sunk to a depth of about 300 feet (November, 1913) on the vein, which dips about 45° southeast. Other development work consisted of about 300 to 400 feet of drifts, a few feet of raises, and a second shaft which had been abandoned but which was said to be about 100 feet deep.

The hanging wall of the vein is in many places fairly well defined, but inasmuch as the foot wall is indistinct and the crosscuts were few, the writer's estimate of 8 to 10 feet as the average width of the deposit is only approximate. The maximum width observed was about 20 feet in a crosscut on the 65-foot level. The vein material consists of what is seemingly an acid eruptive rock, badly shattered and which has been so highly altered that it is difficult to classify. It carries wulfenite, vanadinite, and cerussite, and in many places is heavily stained with iron oxides.

The wulfenite occurs in seams and cracks throughout the ore in thick, tabular crystals, which are commonly one-eighth of an inch and sometimes even an inch in maximum diameter; also as minute specks and irregular masses in the cavities of the rather porous vein material. The wulfenite crystals generally have a bright orange-yellow color, but some are greenish-black to black. Others are bicolored, having black centers and orange-yellow edges, and in some crystals one entire side may be black and the other side orange. The black coloration of these crystals is of economic interest, as it is seemingly due to the presence of finely divided metallic gold, which can be plainly seen in some crystals with the aid of a high-power microscope. The occurrence of native gold in these crystals is discussed in detail on page 115. It suffices here to say that assays made by the author of the orange-colored crystals failed to show even traces of gold, but that those of hand-picked lots of black crystals showed about 140 ounces of gold per ton.

Much of the wulfenite is associated with either cerussite or well-crystallized vanadinite or both; again, these three minerals may occur entirely distinct from one another.

The quantity of wulfenite present is greatly in excess of the vanadinite and cerussite, but no figures as to the average content of the ore in the three different minerals are available, as to the writer's knowledge the mine has never been sampled with the view of determining such data. A 120-pound lot of ore sent to the Bureau of Mines for concentration tests, which was said to be a sample across a 20-foot face on the 65-foot level of the mine, contained 6.08 per cent molybdic trioxide, which corresponds to about 15.5 per cent wulfenite. This sample was undoubtedly much richer in molybdenum than the average run of ore in the mine, which the writer would estimate as probably containing not over 2 or 3 per cent wulfenite.

It is reported that this mine was originally opened for gold, and that on an average the ore contains about one-half an ounce of this metal per ton. Owing to the distance of the property from a water supply sufficient to run a mill, the mine has never been operated to any extent, and at the time of the writer's visit the only ore that had been extracted was that encountered in development. At that time the only equipment at the property was a small dismantled hoist. No pumping had ever been necessary, and the mine was perfectly dry throughout, even at the bottom of the 300-foot shaft.

The wulfenite in the ore is readily concentrated both by wet and dry methods. If, as demonstrated by the concentration tests of the 120-pound sample of ore referred to above most of the gold is contained in the wulfenite, these concentrates should be of high value, as the molybdenum, lead, and gold contained in them can be readily separated and recovered. For a detailed description of the concentration tests made on ore from this mine, see pages 111 to 115.

The writer has been advised that in 1915 this mine was bonded to Col. Epes Randolph and associates, of Tucson, Ariz., who have erected a mill for concentrating the ore and commenced extensive development of the mine.

OTHER OCCURRENCES OF WULFENITE IN ARIZONA.

Besides the wulfenite deposits of the Mammoth, Collins, and Old Yuma mines described above there are many other promising occurrences, particularly throughout the southern part of the State. The Red Cloud, Melissa, and Hamburg mines, which are situated in the Silver mining district, Yuma County, about 80 miles up the Colorado River from Yuma, the nearest point on the railroad, are said to contain a noteworthy quantity of wulfenite in their ores, and have produced many finely crystallized specimens of the mineral. However, these mines have not been operated for many years and have never made any commercial production of wulfenite. Specimens from the Red Cloud and the Hamburg mines show that the wulfenite is often associated with vanadinite. Wulfenite is also reported as occurring at Dome, in the same county, with cerussite in a fluorite gangue, likewise in considerable quantities in several mines in the Castle Dome district, about 30 miles to the north.

In Pima County a property consisting of three and one-half claims owned by Louis Ezekiels, of Tucson, and situated about 25 miles west of that city, is said to show 2 or 3 per cent of wulfenite associated with vanadinite in a well-defined quartz vein. R. O. Boykin, of Tucson, and associates are the owners of a prospect 20 miles from that city and within one-quarter mile of a good wagon road; it is said to show 3 feet of ore carrying 3 per cent of wulfenite associated with vanadinite. Concentration tests are reported to have yielded a product containing about 25 per cent molybdic trioxide. Sufficient water supplies to run a small mill the year round is said to be obtainable from a stream nearby.

According to Pratt[a] there is an occurrence of wulfenite in a mine opened for copper at Troy, in the eastern part of Pinal County, within a few miles of the Gila County line. The wulfenite is said to occur in veins of quartz, which carry 1½ to 3 per cent of the mineral. Pratt further states that, "A concentrating mill has been erected for separating the wulfenite from its gangue, and the Troy-Manhattan Copper Co., which owns the property, is installing a plant for treating the wulfenite concentrates." Inquiries by the writer failed to show that this property had ever become a commercial producer of wulfenite.

J. Jay Sullivan, of Phoenix, Ariz., has recently submitted to the Bureau of Mines a sample of high-grade wulfenite concentrates in which that mineral is associated with considerable vanadinite. The property from which these concentrates were derived is said to be situated 4 miles from Kelvin, in Pima County.

The Mohawk mine, which is only a few hundred yards from the Mammoth mine at Schultz (Pl. VI), is said to show considerable quantities of wulfenite in its workings. At the time of the writer's visit the property had not been operated for many years, and the workings could not be examined, as they were not in condition to risk entry. In the shaft house there were a few tons of wulfenite concentrates which had been recovered from the dump by leasers. The writer would estimate that these concentrates contained 15 to 20 per cent molybdic trioxide. The wulfenite was associated with considerable magnetite.

J. M. Kellog, of Patagonia, Santa Cruz County, owner of the Blue Lead mine 6 miles north of the town, reports that there is a considerable quantity of wulfenite in its ores. The wulfenite is said to be associated with a large amount of pyromorphite.

The Bureau of Mines has received well-cystallized specimens of wulfenite from C. R. Powers, of Gleason, Cochise County. The vein in which this mineral occurs is said to consist of porphyry and to cut a granite formation. The ore shoot, exposed by a shaft 50 feet deep, is reported as being 12 feet wide. The wulfenite is associated with vanadinite and occurs in seams and stringers throughout the ore.

Wulfenite is also said to be a common constituent of the ores of the Tombstone district, Cochise County,[b] and in places it occurs in considerable quantities. In Yavapai County it is reported to occur in the ores of several prospects in the Bradshaw Mountains;[a] also with pyromorphite at the Accidental mine of the Poland Mining Co., of Prescott. The latter property is situated in Lynx Creek Valley near Prescott. The mineral is also said to occur at the Vulture mine in Maricopa County.[b]

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[a] Pratt, J. H., Steel and iron hardening metals: Mineral Resources U. S. for 1903, U. S. Geol. Survey, 1904, p. 342.

[b] Austin, W. L., Silver milling in Arizona: Trans. Am. Inst. Min. Eng., vol. 11, 1882-83, p. 105.

OCCURRENCE OF MOLYBDENITE AT THE LEVIATHAN MINES, MOHAVE COUNTY.

The molybdenite property of the Leviathan Mines Co. is situated in Copper Canyon, Mohave County, on the eastern slope of the Hualpai Mountains. It is in the Cedar Valley mining district, about 3 miles southeast of Copperville and about 25 miles east of Yucca, the nearest point on the Atchison, Topeka & Santa Fe Railway. The property is reached from Yucca by a good wagon road to Copperville, thence by trail down Copper Canyon to the claims, which at their low point in the canyon bottom are at an altitude of about 4,000 feet. The country is arid and as vegetation is confined to desert types no timber is available. Fortunately, water for domestic use can be obtained from springs in the canyon and additional supplies for other purposes from the mine seepage. The wagon road from Yucca to Copperville passes over the main range of the Hualpai Mountains, and reaches an elevation of about a mile. The altitude at Yucca is 1,789 feet, and from this point to the base of the mountains on the west the road has only a slight grade, averaging perhaps 1 or 2 per cent, but on the Yucca side of the range proper the grade is about 7 per cent, and on the Copperville side there are grades as high as 11 per cent. Notwithstanding these steep pitches the road is fairly passable, and auto trucks with loads of 4 to 5 tons make the trip from Copperville to Yucca in about two hours.

The Leviathan properties consist of a group of six claims located on two approximately parallel veins known as the "Whale" and the "Copper Wonder." These veins traverse a granite country rock and consist of white quartz carrying molybdenite and chalcopyrite as the principal ore-forming minerals. The strike of both veins is approximately north-south, and they dip between 85° and 90° west. As the granite country rock is more easily weathered than the hard quartz vein material, erosion has left the veins sticking into the air and made their outcrops easily traceable. In some places the outcrop of the Whale vein is 30 to 40 feet high and its outcrop on the south side of the canyon can be seen for miles. Plate VIII shows views of this outcrop, which can be traced for more than 1,500 feet. The Whale vein which is the larger of the two, varies in width from 6 to 40 feet, and the Copper Wonder vein from about 2 to 20 feet.

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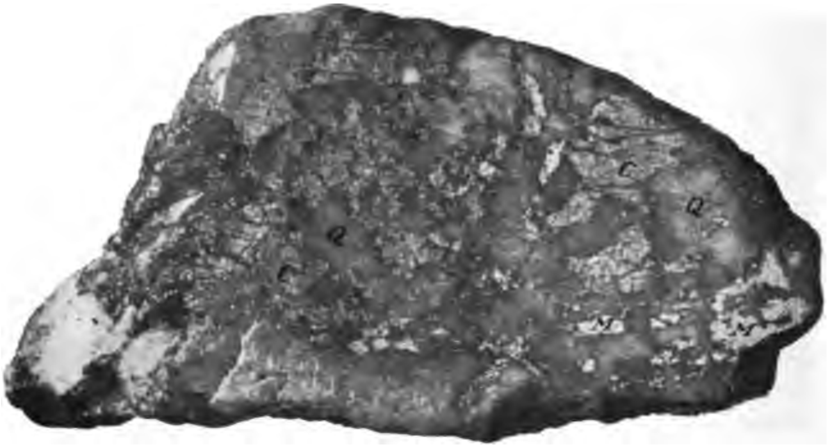
[a] Sanford, S., and Stone, R. W., Useful minerals of the United States: U. S. Geol. Survey Bull. 585, 1914, p. 19.

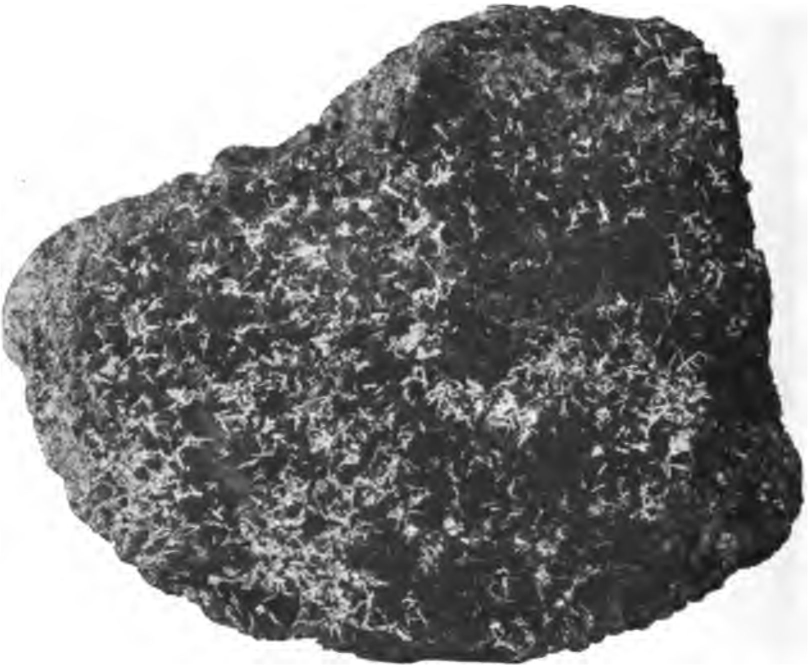
[b] Dana, E. S., A system of mineralogy, 6th ed., 1911, p. 991.

A. NEAR VIEW OF OUTCROP OF WHALE VEIN, COPPER CANYON, NEAR COPPERVILLE, ARIZ., SHOWING ITS HEIGHT.

B. OUTCROP OF WHALE VEIN, LOOKING SOUTH. HEADFRAME OF SHAFT AND PART OF WASTE DUMP ARE SHOWN IN MIDDLE FOREGROUND.



A. SECTION THROUGH TYPICAL ORE FROM WHALE VEIN, COPPER CANYON, NEAR COPPERVILLE, ARIZ. (TWO-THIRDS NATURAL SIZE.) *M*, MOLYBDENITE; *C*, CHALCOPYRITE; *Q*, QUARTZ.



B. SECTION OF TYPICAL MOLYBDENITE ORE FROM LEADER MINE, HELVETIA, ARIZ. (ONE AND ONE-HALF TIMES NATURAL SIZE.)

The molybdenite occurs in amorphous and finely crystalline form in thin veinlets and irregular masses throughout the quartz, and as a fine crystalline powder and as nuggets in vugs and cavities in the veins. Much of it is somewhat intimately associated with chalcopyrite, and in only a few places could the writer obtain specimens that were free from copper. In some places, the individual masses of molybdenite and chalcopyrite are large and are separated by masses of quartz, and in other places they may be very small and closely intermingled. Plate IX, *A*, illustrates a section through a piece of typical ore showing the relation of the two minerals. Where weathering has taken place both the molybdenite and the chalcopyrite have formed alteration products, the former changing to the yellow molybdite, and the chalcopyrite to malachite and azurite. Secondary copper sulphides have also been formed and in several places on the Whale vein native copper occurs. In this vein there are also occasional occurrences of pyrite and small patches of sphalerite, and the ore is reported to carry about 0.02 ounce of gold and 1.4 ounces of silver per ton.

The country rock is a medium-grained gray granite consisting of quartz, feldspar (orthoclase, microcline, and plagioclases varying from albite to labradorite) biotite, muscovite, and small amounts of the usual accessory minerals, zircon and apatite. In the specimen examined there was much zonal development in the feldspar and also much alteration to sericite and possibly kaolin. The rock is more monzonitic in character than the usual granite, but it is a granite nevertheless.

Development work has practically been confined to the Whale vein. At the time of the writer's visit to the property in June, 1915, it consisted of two tunnels driven along the strike of the vein only a few feet above the level of the canyon floor on opposite sides of the gulch, and of a shaft sunk on the north side of the canyon. The tunnel on the south side had been driven for about 210 feet, and that on the north for about 30 feet. The shaft was 100 feet deep and a drift had been driven from the bottom in a southerly direction for 20 feet.

The writer estimates that the average grade of ore exposed in the workings and in the outcrop contains between 2 and 3 per cent MoS_2, and 1½ to 2 per cent copper. This estimate is substantiated by the results of 22 analyses, each representing samples taken over widths of 2 to 17 feet at various points in the tunnel and shaft and on the outcrop. These analyses, which were kindly furnished the writer by R. C. Jacobson, general manager and engineer of the company, averaged 2.73 per cent MoS_2, and 1.71 per cent copper, and indicate that the percentages of molybdenum (metal) and copper in the ore are approximately equal. Several rich streaks in the ore body are reported as containing 5 to 8 per cent MoS_2 over widths of 4 to

6 feet. The ore that is richest in molybdenite appears to contain relatively less copper than that of average grade.

No particular difficulty should be encountered in concentrating this ore either by electrostatic or flotation methods, but the concentrates obtained would consist of a mixture of molybdenite and chalcopyrite, probably in almost equal parts, and in that form their molybdenite content would be of little value. A retreatment of the concentrate by any one of a number of mechanical methods would probably separate the greater portion of the copper from the molybdenite (see pp. 94 to 109), but the writer is of the opinion that none of these processes would completely eliminate the copper from a product containing such a large percentage of it. If such is the case, chemical methods of separation must be employed. The results of a concentration test on this ore by the Wood flotation process are given on page 104.

At the time of the writer's visit to the property, surface equipment consisted of a shaft house containing a Western Iron Works 10-horsepower gasoline hoist, a compressor house containing an Alamo 20-horsepower gasoline engine (type S) belted to a Clayton two-drill air compressor, a blacksmith shop, and a cookhouse. The miners were quartered in tents. The little water seeping into the shaft was removed by means of a small Cameron sinking pump, which delivered it when required to a 3,500-gallon galvanized-iron storage tank. This water supply was used for cooling the engine jackets.

It is estimated that the cost of mining will vary from $1 to $1.50 per ton, according to the scale on which it is carried on. A water supply sufficient for milling purposes could probably be developed and brought to the property at reasonable cost. Moreover, on account of the location of the mine in a canyon having a considerable drainage area, a considerable quantity of mine water will in all probability have to be pumped with further deepening of the shaft and extension of the workings. This water might be used as a supplementary supply for milling. At present the cost of transportation to and from the railroad at Yucca is high, but if a road were built connecting the claims with the road at Copperville this cost would probably not exceed $7 or $8 per ton.

The outcrop of the Whale vein is so exceptionally well defined and so wide and the molybdenite and the copper contents of the vein, as indicated by sampling of the outcrop and workings, are so regular and persistent that the writer considers the property a most promising one notwithstanding the unfortunate association of copper with the molybdenite.

MOLYBDENITE AT THE SMITH & SAWYER AND THE MILLER CLAIMS, MOHAVE COUNTY.

Just below the property of the Leviathan Mines Co. in Copper Canyon and within a distance of 2 miles there are 16 or 17 other well-defined veins, all containing molybdenite associated with chalcopyrite in white quartz, striking in approximately the same direction and having the same general characteristics as those of the Whale and the Copper Wonder veins. Nine claims have been located on these deposits, one group of five claims belonging to Smith & Sawyer, of Kingman, and a group of four owned by J. F. Miller, of Copperville. As far as can be determined from an inspection of their outcrops, the average width of these veins is seemingly 1 to 10 feet and their average content in molybdenite and copper is approximately the same as in the Whale vein, that is, 2 to 3 per cent MoS_2 and 1½ to 2 per cent copper. However, the relative proportions of molybdenite and copper in the different veins seem to vary considerably, but inasmuch as little development work has been done on any of the deposits, this seeming variation may not be true of the veins as a whole but may be solely due to local differences in the individual veins.

The molybdenite in the outcrops of most of the veins has been extensively altered to molybdite, and one of the veins of the Smith and Sawyer group in particular has furnished many remarkably fine crystallized specimens of this mineral. A considerable quantity of bornite was noted in the ore from a vein on the Miller property.

Inasmuch as the mining, concentration, and transportation problems involved in working these veins are identical with those presented by the deposits of the Leviathan Mines Co., they will not be dealt with here.

Considered as a whole, this system of approximately parallel veins on the Leviathan, Smith & Sawyer, and Miller properties constitutes one of the notable molybdenite deposits of the United States.

MOLYBDENITE PROPERTY OF ARIZONA MOLYBDENUM CO., PIMA COUNTY.

The molybdenite property of the Arizona Molybdenum Co. is situated in the northerly end of the Baboquivari Mountains, Pima County, about 35 miles from the railroad at Twin Buttes, the terminal station of a branch line from Sahuarita on the Southern Pacific Railroad. It consists of 10 mining claims of approximately 20 acres each. The ore as represented by a 5-pound sample sent to the writer is seemingly a pegmatite which consists almost entirely of quartz and highly altered feldspar, and throughout which molybdenite, chalcopyrite, and small quantities of galena occur. The ore is reported to contain gold and silver, and a lot said to be an average sample from a width of 8 feet at one point in the vein is stated to have assayed 2.2 ounces of silver, 1.8 ounces of gold, and 3.9 per cent copper. The vein is reported to be 4 to 10 feet wide and to strike southeast. It is said to traverse a coarse-grained granite. Development on the property consists of three shafts, 36, 40, and 60 feet deep, and of about eight smaller shafts each 10 to 12 feet deep. F. J. Wharton, of Tucson, is the president of the company.

MOLYBDENITE AT LEADER MINE, PIMA COUNTY.

The Leader mine, of the Helvetia Copper Co., is situated near the town of Helvetia, Pima County, at an altitude of about 4,700 feet, on the westerly slope of the Santa Rita Mountains, about 27 miles southeast of Tucson. The main tunnel by which the property is opened runs in a general northerly direction, on the contact of an altered limestone with an underlying granite. In operating the mine for copper a number of years ago, an occurrence of molybdenite was encountered at a depth of about 40 feet in a winze sunk from the tunnel level, about 150 feet in from the portal. At the time of the writer's visit to the property, this part of the winze was inaccessible. The following information regarding the occurrence is taken from a description by Schrader and Hill,[a] who visited the mine in 1909.

The winze referred to above is sunk on a quartz vein containing various copper minerals, and dipping at an angle of about 40°. At the tunnel level this vein is only a few inches in width, but there is 1 to 3 feet of mineralized limestone on either side of it, containing chalcopyrite, pyrite, coarse calcite, and quartz, the whole constituting a low-grade copper ore. At a depth of 35 or 40 feet in the winze, molybdenite occurs in both the vein and the surrounding country rock. Eight or ten feet farther down where the dip of the vein flattens the chalcopyrite and pyrite practically disappear, and there is a great increase in the quantity of the molybdenite. At this point there is a body of relatively pure molybdenite ore at least 3 or 4 and perhaps 6 or 7 feet thick. The molybdenite occurs as lenses, irregular bunches, and crystal aggregates associated with both the quartz and the highly altered limestone. Its most common form is as small, flaky crystals.

The rock in which the molybdenite occurs is described as a "dull-brownish and greenish to yellowish mineralized garnetiferous silicified limestone and quartz." It is somewhat massive, and varies from fine to medium grained. It is composed largely of pale-greenish garnet corresponding to grossularite. Quartz is the next most abundant mineral, with calcite, magnetite (or ilmenite), and epidote present in small quantities as secondary minerals.

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[a] Schrader, F. C., and Hill, J. M., Some occurrences of molybdenite in the Santa Rita and Patagonia Mountains, Arizona: U. S. Geol. Survey Bull. 430, 1910, pp. 156-157.

Plate IX, *B*, shows a section through a typical piece of molybdenite ore picked up by the writer from a pile of 2 or 3 tons on the dump at the mine. The molybdenite is seen as small flaky crystals (white) scattered through the groundmass of the highly altered rock.

The ore as represented by the small dump above mentioned contains perhaps 5 to 6 per cent molybdenite. Since the writer's visit to the property some development work has been done on the molybdenite ore and a few tons gotten out. In the annual report of the company for 1915 sales of molybdenite ore during the year are said to have netted $1,891, and it is stated that there were approximately 100 tons of ore in sight that should net about $26 a ton.[a]

OTHER OCCURRENCES OF MOLYBDENITE.

It is reported that a few tons of rich molybdenite ore was taken out several years ago by the Cuprite Copper Co., of Tucson, in mining a large pocket of copper ore on its property in the northwest end of the Santa Rita Mountains, in Pima County. The property is about 8½ miles south of Vail, the nearest point on the Southern Pacific Railroad, and at an altitude of about 4,000 feet. No molybdenite ore in place could be found by the writer in a hasty examination of the mine workings, but several specimens containing 10 to 20 per cent of the mineral were picked up on the mine dump. In these the molybdenite was associated with chalcopyrite and copper carbonates in a gangue of highly altered and silicified limestone. No evidence as to the form of the molybdenite deposit or the possibilities of further occurrences of the mineral could be obtained.

E. O. Stratton, of Tucson, and associates are the owners of a molybdenite property situated on Marble Creek, in the Santa Catalina Mountains, in Pima County, about 20 miles northwest of Tucson and 15 miles south of Oracle. Some promising showings of molybdenite are said to have been encountered in prospect work.

Other occurrences of molybdenite in Pima and Santa Cruz Counties are as follows:

At the Ridley mine, owned by C. B. Ridley, of Helvetia, and situated in the foothills about 1½ miles southwest of that town.

At the McCleary prospects in Madera Canyon, about 10 miles south-southwest of Helvetia and 35 miles south of Tucson.

In the foothill part of Providencia Canyon, about 10 miles northeast of Nogales and 5 miles north of the United States-Mexican boundary line near the Golden Rose and at the Buena Vista mine, the latter owned by the Banco del Oro Mining Co., of Magdalena, Sonora, Mexico.

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[a] Editorial Eng. and Min. Jour., vol. 101, 1916, p. 482.

At Duquesne, about a quarter of a mile west of the Belmont mine, on property owned by Capt. O'Connor, of Duquesne.

At the Benton mine and the Line Boy prospect in San Antonio Canyon, about 2 miles south of Duquesne and 15 miles north-northeast of Nogales. The Benton mine is owned by Dennis Coughlin and partners, and the Line Boy prospect by Capt. O'Connor, all of Duquesne.

In all these localities the molybdenite occurs in granite or in quartz traversing the granite. None of the properties has been developed beyond the prospect stage, and present showings do not indicate that any of them are of commercial importance. The above information concerning these occurrences was obtained from detailed descriptions by Schrader and Hill.[a]

It is stated [b] that one of the mines of the Santa Nina Co., of Patagonia, has a 6-inch or 7-inch vein of high-grade molybdenite ore in one of their copper mines.

Molybdenite is found in the ores of the Clifton-Morenci district, Greenlee County, in fissure veins with pyrite, chalcopyrite, and sphalerite.[c] It also occurs in the disseminated ores at Miami, Gila County, in the primary ores of the chloride district, Mohave County, in a granito-gneiss, and as an original mineral in the ores at the Ray and Kelvin mines in Pinal County.[d]

CALIFORNIA.

Although there has never been any commercial production of molybdenum ore in California, molybdenite is widely distributed throughout the State and development work on some of the known occurrences of the mineral will probably prove them to be of economic importance.

Following is a list of deposits that have come to the author's attention. The list includes 41 occurrences of molybdenite in 16 different counties. Brief notes regarding six of these occurrences follow. Wulfenite has been reported in a few localities, principally in the southern part of the State, but as far as known to the writer none of the deposits of this mineral is of commercial interest.

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[a] Schrader, F. C., and Hill, J. M., Some occurrences of molybdenite in the Santa Rita and Patagonia Mountains, Arizona: Contributions to economic geology: U. S. Geol. Survey Bull. 430, 1910, pp. 154-162.

[b] Private communication from J. M. Kellogg, Patagonia, Ariz.

[c] Lindgren, W., The genesis of the copper deposits of Clifton-Montmorenci, Arizona: Trans. Am. Inst. Min. Eng., vol. 34, 1905, pp. 515, 523.

[d] Sanford, S., and Stone, R. W., Useful minerals of the United States: U. S. Geol. Survey Bull. 585, 1914, p. 16.

Occurrences of molybdenite in California.

Location.	Remarks.	Reference.
Calaveras County:		
New Big Tree	Associated with feldspar	Specimen in U. S. National Museum.
Zeila mine	Associated with quartz, sericite, and pyrite.	Do.
Eldorado County:		
Fairplay	Small crystals with calcite, chalcopyrite, garnet, and epidote.	Specimen in Agassiz Museum.
Near Red Hill, 9 miles east of Sanger.	Flakes up to ½ inch in diameter in white quartz.	Specimens in collection of U. S. Geol. Survey.
36 miles northeast of Millwood.	Fair-sized flakes in white quartz.	Specimen in California Bureau of Mines Museum.
Grizzly Flat	Large flakes	Do.
Fresno County:		
Kings River Canyon, copper mine.	Flakes in quartz	Do.
Green Mountain, south fork of San Joaquin River.		Do.
Fresno Flat		Do.
Buchanan prospect, west of Granite Creek, south fork of Kings River.	See p. 62.	
Near Soda Springs	In quartz	Do.
Inyo County:		
Bishop, near Hillside Dam, south of Bishop Lake.	See p. 61.	
White Mountains		Do.
West Arm of Death Valley, south of Lida.		Eng. and Min. Jour., vol. 81, 1906, p. 205.
Kern County:		
Gold region near Havilah		U. S. Geol. Survey Bull. 585, 1914, p. 33.
Near Caliente	See p. 63.	
2½ miles northeast of Randsburg.	Deposit said to be 30 feet wide and traced for 2 miles on lime-schist contact.	Eng. and Min. Jour., vol. 96, 1913, p. 186.
Los Angeles County:		
Northeast part of county		Mineral Resources U. S. for 1901: U. S. Geol. Survey, 1902, p. 266.
Los Angeles	Associated with feldspar and quartz.	Specimen in U. S. National Museum.
Mono County:		
12 miles northwest of Bridgeport, on west fork of Walker River.	Molybdenite-bearing core about 20 feet wide in quartz vein associated with molybdite.	Mineral Resources U. S. for 1901: U. S. Geol. Survey, 1902, pp. 265-266.
Mono Lake	Crystals on quartz	Specimen in Agassiz Museum.
Cameron, near Bridgeport		Specimen in California Bureau of Mines Museum.
Minnie mine, Sweetwater Range.	In quartz	Do.
Nevada County:		
Mariposa mine, Rosario district.		Do.
Mayflower mine, Nevada City.	Flakes up to 2 inches in diameter in quartz.	Do.
Placer County:		
Golden Stag mine, Ophir region.	Small flakes	U. S. Geol. Survey Bull. 585, 1914, p. 33.
On Red Mountain, 3 miles north of Cisco.	Crystalline molybdenite in white quartz.	Specimens in collection of U. S. Geol. Survey.
Riverside County:		
4½ miles northeast of Corona	See p. 62.	
About 17 miles west of Hemet, in sec. 3, T. 5 S., R. 2 E.	Fair sized flakes in iron-stained quartz.	Do.
16 miles from Corona	In siliceous gangue	Mineral Industry, vol. 7, 1898, p. 514.
San Bernardino County:		
Leastalk	Fine grains in white quartz with chalcopyrite.	Specimens in collection of U. S. Geol. Survey.
San Diego County:		
40 miles east of San Diego	See p. 60.	
Near Dulzura	Small flakes with quartz and ferro-magnesium minerals.	Specimens in collection of U. S. Geol. Survey.
Campo	In granite	Specimen in California Bureau of Mines Museum.
Do.	Associated with feldspar and quartz.	Specimen in U. S. National Museum.
Dewey mine at Grapevine, near Warners Hot Springs.		Mineral Industry, vol. 11, 1902, p. 477.
Shasta County:		
Tom Neal Mountain, 5 miles from Delta.	In granite	Specimen in California Bureau of Mines Museum.
Hazel Creek	do	Do.
Lamoine		U. S. Geol. Survey Bull. 585, 1914, p. 33.

Occurrences of molybdenite in California—Continued.

Location.	Remarks.	Reference.
Tulare County:		
Three Rivers		Specimen in California Bureau of Mines Museum.
California Hot Springs	See p. 62.	
Tuolumne County:		
West of Tower Peak	In quartz gangue on a contact of marble and a white schistose pyroxene with epidote and sphalerite.	Turner, Am. Jour. Sci., vol. 5, 1898, p. 427.
South of Knight Creek, northeast of Columbia.	In granite	Do.
Ventura County:		
On Alamo Mountain, northeastern part of county.	Vein said to be 8 to 15 feet wide.	Mineral Resources U. S., 1901, U. S. Geol. Survey, 1902, pp. 265-266; Eng. and Min. Jour., vol. 72, 1901, p. 337.

MOLYBDENITE AT PROPERTY OF SANTA MARIA MOLYBDENUM MINING & MILLING CO., SAN DIEGO COUNTY.

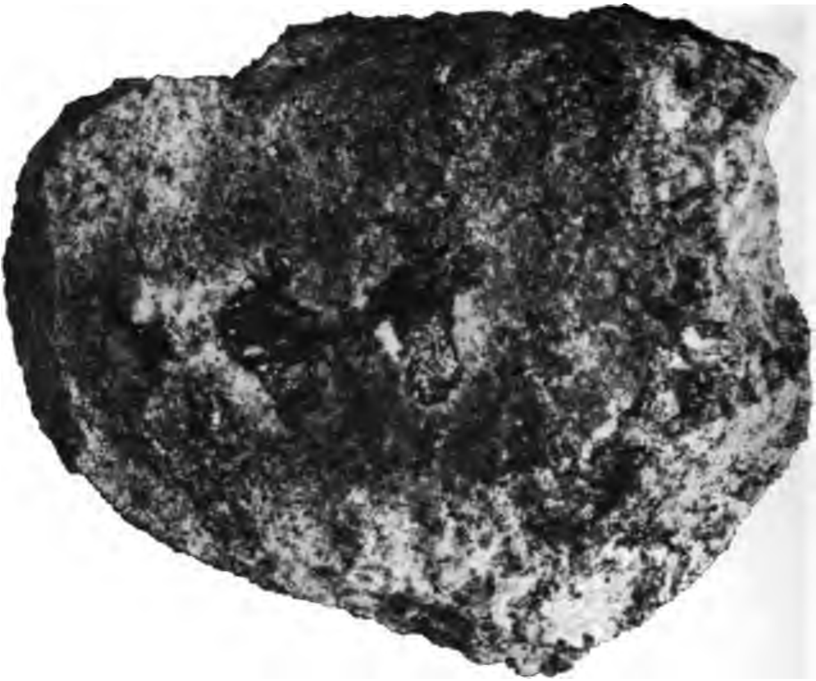
The property of the Santa Maria Molybdenum Mining & Milling Co., of San Diego, is situated about 40 miles east of that city in the hills to the southeast of the San Pasquale valley and is readily reached from San Diego by automobile, the road being excellent. It is understood to consist of 160 acres of patented ground.

The molybdenite-bearing deposit consists of a large granite dike varying in width from 35 to 100 feet and striking in a general northeasterly direction. The outcrop of this dike is about half a mile long and is particularly well defined. Plate X, *A*, shows a view looking toward the northwestern end, where the outcrop terminates in an almost perpendicular cliff about 200 feet high. The canyon on which this cliff faces drains into San Pasquale valley which may be seen in the distance in Plate X, *A*. A good idea of the size of the deposit may be gained from a comparison with the size of the persons shown in the illustration.

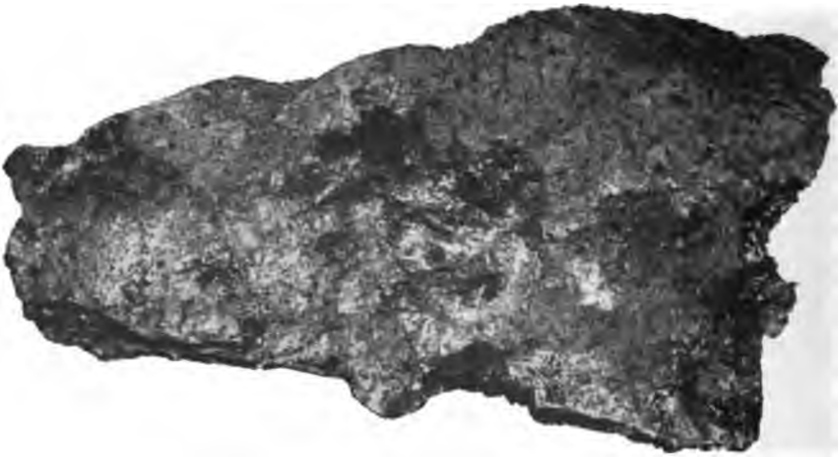
The molybdenite occurs sparingly throughout the granite in crystal aggregates and irregularly shaped masses of radial structure, most of which vary in size from one-eighth to 1 inch in maximum diameter. It is associated with a little pyrite. Both the molybdenite and the pyrite have been altered, the former slightly and the latter extensively. In a few instances the transformation of the molybdenite to molybdite has been complete, the latter mineral crystallizing in radial groups, such as are shown in Plate V, *B*, which illustrates a specimen of molybdite from this property. More often, however, the molybdenite has been only partly altered, resulting in many interesting specimens, one of which is pictured in Plate II, *B*. The iron oxide formed by the alteration of the pyrite has stained the larger part of the outcropping granite a light red. In this stained part the molybdenite areas are almost invariably surrounded by a narrow rim of unstained rock, as is rather clearly illustrated in Plate

A. OUTCROP OF DIKE ON SANTA MARIA PROPERTY, SAN DIEGO COUNTY, CAL., LOOKING NORTHWEST.

B. OUTCROP OF LOW-GRADE MOLYBDENITE ORE AT PRIMOS MINE, NEAR EMPIRE, COLO.



A. MOLYBDENITE IN IRON-STAINED GRANITE FROM SANTA MARIA PROPERTY, SAN DIEGO COUNTY, CAL. (FOUR-FIFTHS NATURAL SIZE.)



B. MOLYBDENITE IN GRANITE FROM JOHN FLETCHER QUARRY, NEAR CORONA, CAL. (TWO-THIRDS NATURAL SIZE.)

XI, *A*, which shows a typical specimen of molybdenite ore from the property.

The granite of which the dike is composed is medium grained, and where not iron stained it is light gray to almost white. A microscopical examination of it shows the presence of the following minerals: Quartz, orthoclase, plagioclases varying from albite to oligoclase, biotite, a few minor accessory minerals, and pyrite.

The only development work on the property at the time of the writer's visit in 1914 was five or six open cuts partly crosscutting the dike. Some of them showed ore which might average from 0.25 to 0.5 per cent molybdenite, but from all indications the dike as a whole contains a very much smaller percentage of the mineral. Unless further development work discloses areas richer in molybdenite, the writer does not believe that the occurrence will prove of economic importance. However, as the character of the ore is such that the molybdenite can be readily concentrated into a high-grade product, the discovery of richer ore might lead to successful operations.

A lot of ore weighing 313 pounds, kindly shipped the Bureau of Mines by the owners of the property for concentration tests, and said to be unsorted, assayed 0.47 per cent MoS_2. For the results of hand-picking and concentration tests of this ore, see pages 97 to 99.

MOLYBDENITE NEAR BISHOP, INYO COUNTY.

Specimens of molybdenite associated with molybdite in white quartz have been submitted to the bureau by M. C. Hall, of Bishop. The deposit from which the ore came is said to be situated at an altitude of about 10,000 feet in the Sierra Nevada Mountains about 20 miles from Laws, the nearest station on the Southern Pacific Railroad. There is a road from Laws passable by automobile to within a mile and a half of the property, thence a poor wagon road for a mile, but no road for the last half mile. It is stated, however, that one could readily be built to cover the remaining distance. The outcrop of the deposit is reported to be about 100 feet long and the same distance across. Practically no development work has been done. In a 40-pound sample said to represent average ore, the author would estimate the molybdenite content to be 1 to 2 per cent. With the exception of the occasional occurrence of small grains of chalcopyrite, no minerals other than molybdenite and its alteration product, molybdite, were noted in the quartz. The molybdenite occurs in flakes varying in size from minute specks to an eighth of an inch or more in maximum diameter, and the ore is seemingly of such a nature as to be readily concentrated by either electrostatic or flotation methods. Development work may perhaps prove the deposit to be of economic importance.

MOLYBDENITE AT THE BUCHANAN PROSPECT, FRESNO COUNTY.

Specimens of molybdenite ore have been submitted to the bureau by Paul Buchanan, of Kearney Park, Cal. They are said to have come from a deposit situated on the north side of the south fork of Kings River, between Granite and Copper Creeks, and about 2 miles west of Kanawyer's Camp, in T. 13 S., R. 31 E. The property is difficult of access, being 25 miles or more from the nearest wagon road at Hume, which in turn is a greater distance from the nearest railroad station. The vein in which the molybdenite occurs is reported to have an average width of 1 to 3 feet and to be traceable on the surface for about 3,000 feet, but the outcrop carries molybdenum for only about 2,000 feet. The strike of the mineralized part of the vein is said to be almost north and south. The specimens submitted consist of granite cut by small quartz stringers occasionally containing flakes of molybdenite up to one-fourth inch in maximum diameter. The molybdenite is associated with a small quantity of chalcopyrite. The granite consists of quartz, feldspar, and biotite, and is somewhat iron stained. If the average width of the vein is only 1 to 3 feet, as stated, and the grade of the ore is not richer than the samples submitted, the inaccessibility of the property precludes the possibility of its commercial importance.

MOLYBDENITE NEAR CORONA, RIVERSIDE COUNTY.

An occurrence of molybdenite about 4½ miles northeast of Corona in a granite quarry operated by John Fletcher has been noted by Hess.[a] Specimens submitted to the bureau show the mineral to occur in a medium-grained gray granite in flakes up to an inch in diameter. A piece of typical ore is illustrated in Plate XI, *B*. Hess, who visited the deposit in 1907, states that: "The granite is cut by thin pegmatite dikes one-half inch to 2 inches wide. Molybdenite in flakes up to one-half inch across accompanies the dikes in small quantity. In one or two places small flakes may be found in the granite for a distance of 2 or 3 inches from the dikes. The only other metallic mineral found in the dike is iron pyrites in which an assay is said to have shown some copper, gold, and silver." The deposit is said to be small and is probably not of economic importance.

MOLYBDENITE NEAR CALIFORNIA HOT SPRINGS, TULARE COUNTY.

Molybdenite in small flakes up to about one-fourth inch maximum diameter occurs in milky quartz in a large vein about one-half mile from California Hot Springs. This town is about 22 miles west of Ducor, the nearest point on the Southern Pacific Railroad, from which it is reached by a good automobile road. The deposit is situated in sec. 31, T. 23 S., R. 31 E., at an elevation of about 3,300 feet, and is owned by L. S. Wingrove, of California Hot Springs. Practically no development work has been done on the vein, but a hasty examination of a considerable part of the outcrop indicated to the writer that the occurrence of molybdenite, even as minute specks, is so occasional that there seems to be no possibility of the deposit being of commercial importance.

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[a] Hess, F. L., Some molybdenum deposits of Maine, Utah, and California; Contributions to Economic Geology: U. S. Geol. Survey Bull. 340, 1907, p. 238.

MOLYBDENITE AT CALIENTE, KERN COUNTY.

J. B. Ferris of Caliente has submitted to the bureau specimens of low-grade gold ore carrying molybdenite in finely disseminated grains, coming from the Golden group of claims a few miles south of the town. The ore body is large, but as determined by samples taken by the author from several places in the tunnels and the drifts by which it is developed, it is so poor in molybdenite as to probably be of no commercial importance as a source of that mineral.

COLORADO.

Molybdenite is widely distributed throughout Colorado, its occurrence having been noted in almost every mountain county. The principal deposits visited by the author are at Red Mountain near Empire in Clear Creek County, and near Climax and Breckenridge in Summit County. A description of these deposits, together with notes as to some of the other occurrences listed in the accompanying table, are given in the following pages.

Several occurrences of wulfenite in the State have been reported, but so far as known these are only of mineralogical interest.

Occurrences of molbydenite in Colorado.

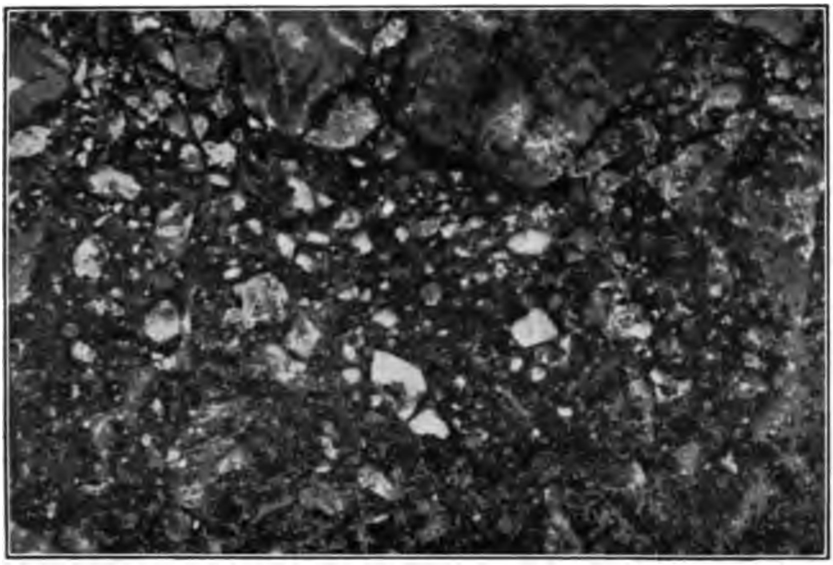
Location of deposit.	Remarks.	Reference.
Chaffee County:		
Salida	In quartz	Specimen in Colorado State Museum.
South of Fisher	See p. 70	
Near Buena Vista	See Pl. II, *A*	Do.
Clear Creek County:		
Red Mountain near Empire	See p. 64	
Conejos County:		
At Platoro	In spherical forms with concentric structure.	Dana, E. S., A system of mineralogy, 6th ed., 1911, p. 1042.
Custer County:		
Grape Creek	In decomposed basic rock with calcite, chlorite, and pyrite.	Specimen in U. S. National Museum.
12 miles north of Westcliffe	See p. 71	
Eagle County:		
Near Redcliff		
Fremont County:		
Near Rito Alto Peak	See p. 70	
7 miles south of Parkdale	See p. 70	
Gilpin County:		
From Apex	With molybdite	Specimen in Colorado State Museum.

Occurrences of molbydenite in Colorado—Continued.

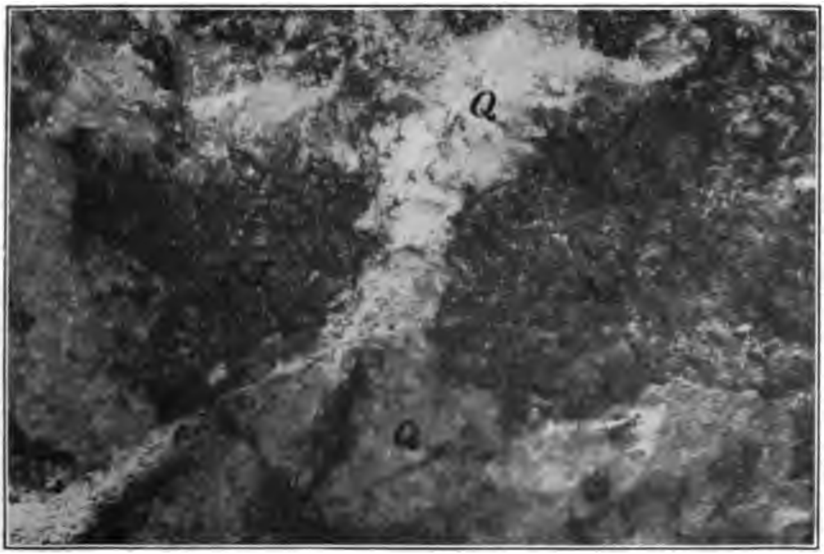
Location of deposit.	Remarks.	Reference.
Gunnison County:		
4 miles north of Pitkin near Rock Creek.		Mineral Industry, vol. 6, 1898, p. 485.
At the head of Crystal River near Marble.		Letter from J. C. Hersey, Leadville, Colo.
Near Tin Cup	Crystalline molybdenite in white quartz.	Specimens in collection of U. S. Geol. Survey.
2 miles from Pitkin	In quartz veins	U. S. Geol. Survey Bull. 585, 1914, p. 46.
8 miles southeast of Marble	See p. 71	
Huerfano County:		
2 miles southeast of Mosca Pass.	Large flakes in float	Letter from John O'Toole, Denver, Colo.
Lake County:		
In Maid of Erin mine at Leadville; also near the head of Big Evans Gulch.		Eng. and Min. Jour., vol. 80, 1905, p. 127.
Larimer County:		
4 miles south of St. Cloud	See p. 72	
Ouray County:		
Engineer Mountain		Letter from E. C. Weatherly, Ouray, Colo.
Pitkin County:		
South of Red Mountain	See p. 71	
In Lincoln Gulch southeast of Roaring Fork of the Grand River.	In silicious gangue	Letter from J. C. Hersey, Leadville, Colo.
San Juan County:		
Between Maggie and Picayune Gulches and Red Peak.	With copper, nickel, and bismuth minerals.	Trans. Am. Inst. Min. Eng., vol. 21, pp. 189-190.
Near Silverton		U. S. Geol. Survey Bull. 585, 1914, p. 46.
San Miguel County:		
1 mile east of Ophir	See p. 69	
Summit County:		
On Bartlett Mountain	See p. 68	
On Chalk Mountain	See p. 68	
Fremont Pass		U. S. Geol. Survey Bull. 585, 1914, p. 46.
Near Kokomo	See p. 72	
2 miles north of Kokomo	See p. 72	
Lenawee mine, between Kingston and Montezuma.		Specimen in courthouse, Breckenridge.
Southern slope of Quandary Mountain.	See p. 67	
Teller County:		
Cripple Creek		

MOLYBDENITE MINE OF PRIMOS CHEMICAL CO., NEAR EMPIRE.

The molybdenite mine of the Primos Chemical Co., near Empire, Clear Creek County, consists of a group of nine claims situated on the eastern slope of Red Mountain, at an altitude of about 10,500 to 12,000 feet. The property is reached from Empire, a station on the Georgetown branch of the Colorado & Southern Railroad, by a good wagon road 14 miles long. The ore-bearing bodies consist of three veins of low-grade ore, the general strike of which, as far as determined by present development work, lies between east by west and northeast by southwest, across the ridge of the mountain. The strikes of the individual veins show a convergence in a westerly direction. Their dip, as indicated by the position of the outcrops with reference to their intersection by a tunnel several hundred feet below, is 50° to 60° in a general northwestern direction. The veins are named from the southeast, and are known as Veins No. 1, No. 2, and No. 3. The ore zone in general; that is, the ground included



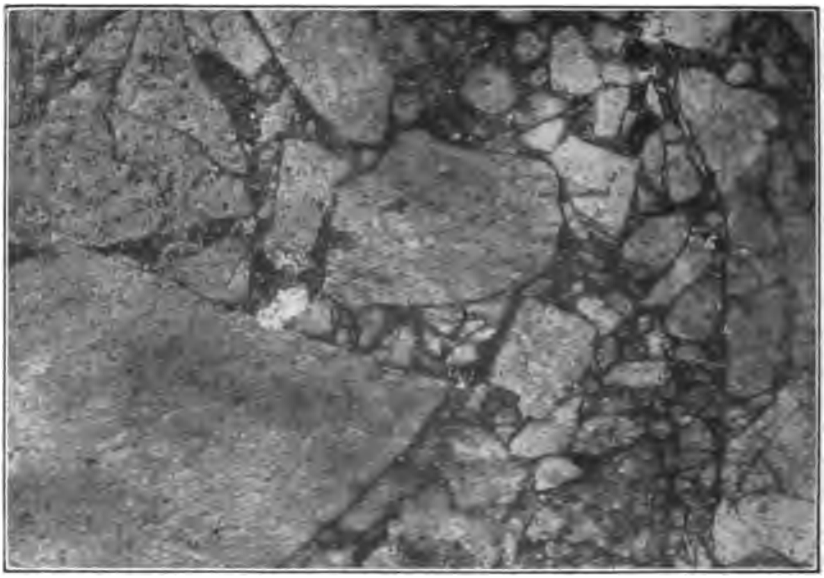
A. SECTION THROUGH TYPICAL MOLYBDENITE ORE FROM PRIMOS MINE, NEAR EMPIRE, COLO. (NATURAL SIZE.)



B. TYPICAL MOLYBDENITE ORE FROM BARTLETT MOUNTAIN, SUMMIT COUNTY, COLO. (NATURAL SIZE.) *M*, MOLYBDENITE; *Q*, QUARTZ.

A. VIEW OF RED MOUNTAIN, NEAR EMPIRE, COLO.

The arrow indicates the waste dump at the mouth of the upper tunnel of Primos Mine.



B. SECTION THROUGH TYPICAL MOLYBDENITE ORE FROM GREAT WESTERN MINE, NEAR CHICO, PARK COUNTY, MONT.

between the footwall of Vein No. 1 and the hanging wall of Vein No. 3, where cut by the tunnel, is about 200 feet wide. The veins vary greatly in width, both among themselves and along their strikes. Further, they are not particularly well defined, the ore running off into the walls in the form of thin veinlets and stringers.

Vein No. 1 has not been sufficiently developed to warrant even an approximate statement of its average width, but at its intersection by the upper tunnel it is 3 to 4 feet across. Vein No. 2, as exposed by about 1,000 feet of development work, shows a width varying from 3 or 4 inches to 4 or 5 feet, and it has an average width of perhaps 2 feet. Vein No. 2, at its intersection by the upper tunnel, showed 10 feet of ore.

The ore occurs in two forms—one consists of an alaskite (?) breccia in which the molybdenite occurs as occassional small flakes but more often in finely granular form associated with iron pyrite in the interstices of the breccia; in the other the molybdenite occurs in small veinlets and stringers running off into the alaskite-porphyry (?) country rock. The brecciated type of ore constitutes the vein material proper, and is well illustrated by Plate XII, *A*. Oxidation of the molybdenite along the outcrops of the veins has resulted in the formation of considerable molybdite, but the latter mineral disappears entirely with depth. It is stated that the ore contains no copper or other deleterious elements.

The author first visited the property in July, 1913, when development work consisted of only two or three shallow open cuts along the outcrop of one vein. The existence of ore bodies was evidenced, however, by large masses of vein material exposed along the outcrops and by the great quantities of float found throughout the rock slides on the mountain slope below. One of these large masses of outcropping vein material, which showed considerable molybdenite here and there over its entire surface, and was colored in many places by yellow molybdite, is pictured in Plate X, *B*.

Development of the property was started in earnest in November, 1914, when the present owners obtained control, and to August, 1915, work had been pursued at the rate of about 350 feet per month. At the latter date development consisted of an upper working with 2,500 to 3,000 feet of tunnel, drifts, and crosscuts at two-thirds the distance to the top of the mountain and at an altitude of about 11,800 feet; and also of a tunnel which had just been commenced about 600 feet lower down. The situation of the upper workings is well shown in Plate XIII, *A*.

The upper workings consist roughly of a main tunnel running in a northwesterly direction for about 500 feet, and of a drift along Vein No. 2 about 800 feet west. Twenty-one crosscuts up to 20 feet in length have been made along this drift, usually at intervals of about 30 feet, but occasionally at 15-foot intervals. A stope has been started on this vein, and in July, 1915, it had been opened for a horizontal distance of about 40 feet and mined to a height of about 20 feet, the ore having an average width of about 4½ feet. A second and larger stope has been begun to the north of Vein No. 2. The ore body at this point is thought to be either separated from the vein by a large "horse," or to lie on the intersection of veins No. 2 and No. 3. This stope has been mined for a horizontal distance of about 130 feet and to a height of 40 to 45 feet; in width it varies from 6 to 15 feet.

Only a few feet of work has been done on vein No. 1 as the showing of ore encountered at its intersection by the tunnel was poor. Vein No. 3 has been drifted on for about 20 feet on either side of the tunnel, and 10 feet of good ore is said to have been encountered in these drifts.

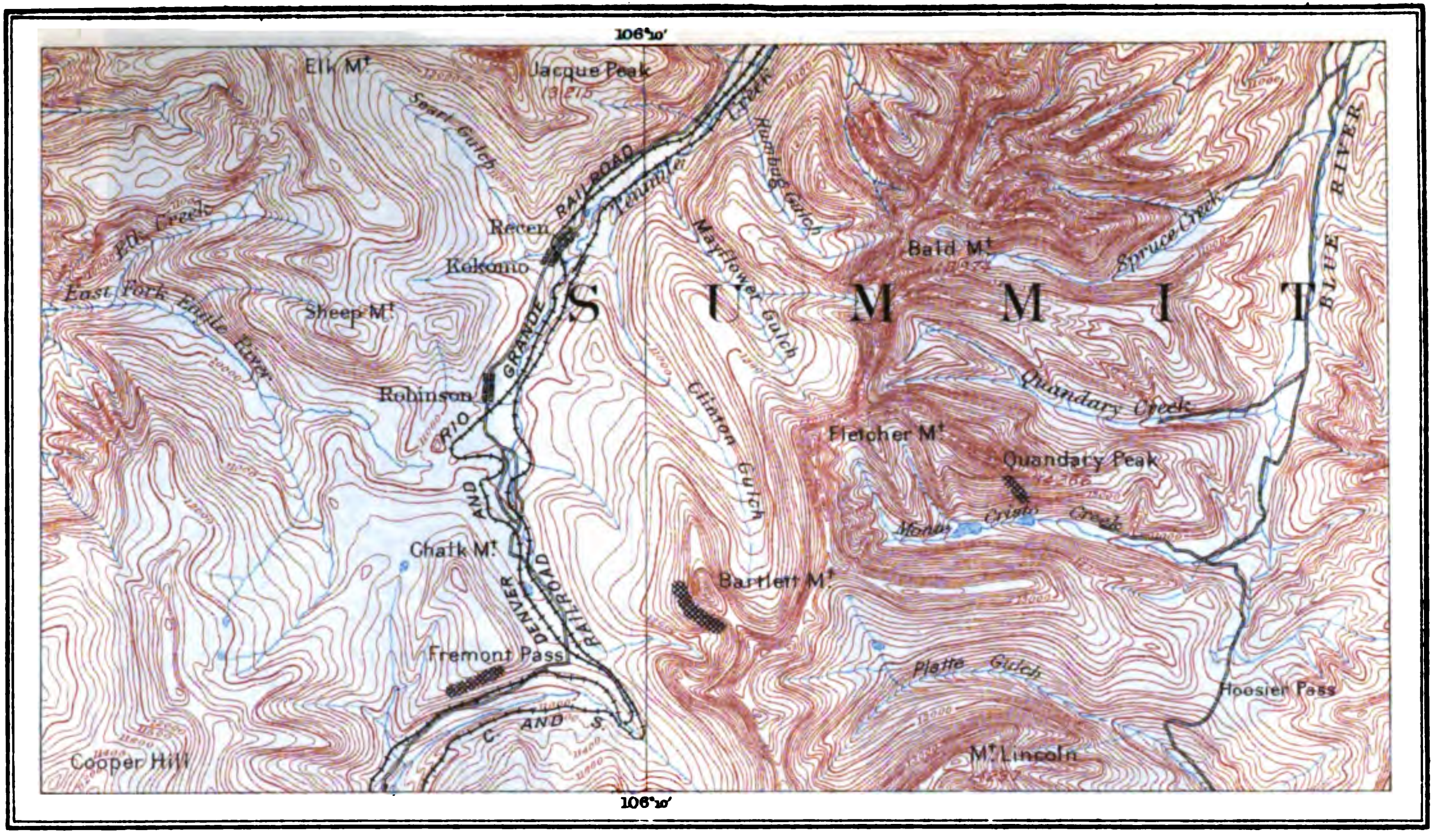
As already stated, work on the lower tunnel was commenced in August, 1915. Assuming that the dip of the veins is 55° and that they will be encountered on this level, over 1,000 feet of backs will be developed by this lower tunnel.

Stoping is carried on largely by hand, as the ground is fairly soft and easily worked. Air drills, however, are used in running the tunnels and drifts.

The ore is trammed to a sorting house at the mouth of the upper tunnel and dumped into a bin from which it is drawn by the sorters as needed. Ore from the bin is screened on small 2-mesh hand sieves. The undersize through the screens is saved and the oversize hand sorted. The rejected oversize is thrown into a car which carries it to the waste dump, and the selected ore is sacked. Filled sacks weigh 75 to 125 pounds each, according to the size and richness of the ore. These are packed down the mountain on mules and stacked near the wagon road. At the time of the author's visit, in July, 1915, the pack train consisted of 14 animals, each of which carried three to four sacks of ore. At the base of the mountain the sacks are loaded onto wagons, weighed, and hauled to the railroad station at Empire, where their contents are emptied into freight cars for shipment to the Eastern plant of the company. From January 1, 1915, to August, 1915, shipments are reported to have averaged two to three carloads of 22½ tons each per week.

The mine has not been in operation for sufficient time and the ore bodies are not well enough developed to justify any statements as to mining cost.

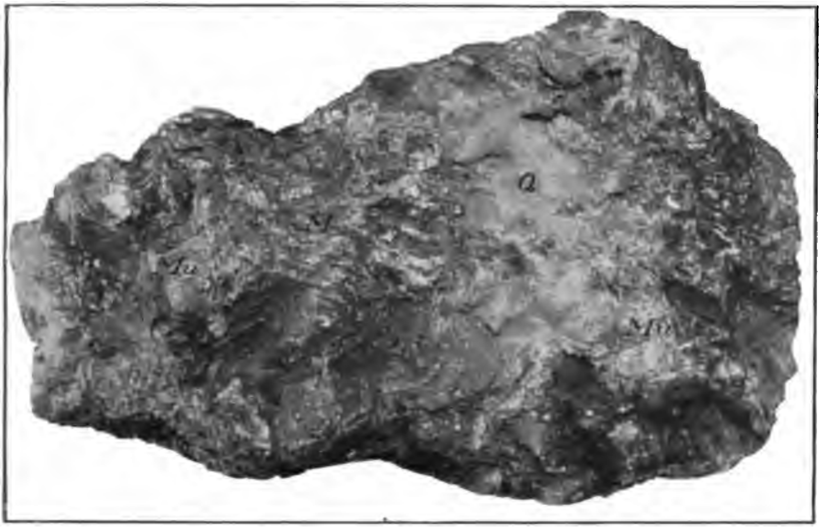
A combined bunk and compressor house, which also contains an office, dining-room and quarters for cooking, is situated about 1,300 feet below the mouth of the upper tunnel and close to the wagon



MAP OF PART OF SUMMIT COUNTY, COLORADO,

SHOWING LOCATION OF MOLYBDENITE DEPOSITS ON QUANDARY, BARTLETT, AND CHALK MOUNTAINS

Scale $\frac{1}{125000}$ 1 ½ 0 1 2 3 4 5 Miles Contour interval 100 feet.



A. TYPICAL MOLYBDENITE ORE FROM SALAMANDER CLAIM, QUANDARY PEAK, SUMMIT COUNTY, COLO. (TWO-THIRDS NATURAL SIZE.) *M*, MOLYBDENITE; *Q*, QUARTZ; *Mu*, MUSCOVITE.

B. SCRAPING TAILINGS FOR RETREATMENT TO RECOVER WULFENITE, MAMMOTH, ARIZ.

road. The compressor room in this building contains a 60-horsepower boiler which is fired with wood; it supplies steam for the operation of an Ingersoll-Rand "Imperial" air compressor, type 10. The two steam cylinders on the compressor are 13 by 10 inches and 8 by 10 inches, and the corresponding air cylinders are 14 by 10 inches and 7½ by 10 inches. Although it is rated as a three-drill machine it will operate only two drills at this altitude.

Wood for the boiler is obtained by cutting standing timber in a burned-over area of forest in the vicinity. It is hauled to a small sawmill situated a few hundred yards south of the boiler house, and cut into 4-foot lengths. Stables have been built close to the bunk and compressor house. At the upper tunnel level there is a bunk house and blacksmith shop and the ore-sorting house already referred to.

The production of this property to date has been probably 1,500 to 2,000 tons of ore, averaging approximately 2 per cent molybdenum. In opening the property during the winter months many difficulties were encountered, owing to the altitude and to winter storms.

SALAMANDER AND BLUE VALLEY MOLYBDENITE CLAIMS, NEAR BRECKENRIDGE, SUMMIT COUNTY.

There is an interesting occurrence of molybdenite about 11 miles southwest of Breckenridge, Summit County. The deposit is on the south slope of Quandary Mountain at an altitude of about 12,000 feet (see Pl. XIV), and consists of two pegmatite veins which contain the molybdenite, and on which are two claims known as the Salamander and the Blue Valley. These claims are reached from Breckenridge by a good wagon road for 9 miles up the valleys of Blue River and Monte Christo Creek, thence continuing up the valley of the latter stream for 1½ miles by a poor mountain road, and then across the creek and up the mountain side for half a mile by trail.

At the time of the author's visit to the deposit in October, 1913, a violent snowstorm was in progress, and for that reason his examination was necessarily incomplete.

The pegmatite veins already referred to consist largely of muscovite and quartz with some feldspar, and carry molybdenite and chalcopyrite as accessory minerals, as well as small quantities of molybdite developed by alteration of the molybdenite in places where the latter has been exposed to the air for a considerable time. The veins, which appear to be nearly vertical, traverse a dark-gray micaceous schist or gneiss composed essentially of quartz, muscovite, biotite, and feldspar, and strike in a general northerly direction.

A specimen of typical ore from the Salamander claim is illustrated in Plate XV, *A*. A sample of about 100 pounds of picked ore, which the author selected as representative of the general grade of concentrating ore that this claim could produce, on analysis showed 4.35

per cent molybdenite and 0.45 per cent copper. The molybdenite occurs in coarsely crystalline aggregates up to 1½ inches in maximum diameter, but on account of its association with chalcopyrite and with such a large percentage of muscovite, it probably could not be recovered in a high-grade concentrate without a rather complex treatment.

On the Salamander claim a tunnel has been run for a distance of 20 feet on the vein, which at this point has a width of 3 to 4 feet.

On the Blue Valley claim there is only a small showing of molybdenite, and no development other than the putting in of several shots at various places along the outcrop. The vein averages only 4 to 6 inches in width and gives no promise of any commercial production of ore.

Directly down the mountain from the claims there is a small lake, and one-half mile farther up the gulch a larger lake at an elevation of 300 or 400 feet above the one first mentioned. Ample water power for all mining purposes could easily be developed from this upper lake, and there is sufficient timber in the immediate neighborhood for all prospective mining needs.

MOLYBDENITE ON BARTLETT MOUNTAIN, SUMMIT COUNTY.

There is a large low-grade deposit of molybdenite ore above timber line at an elevation of approximately 12,000 feet on the southwestern slope of Bartlett Mountain, Summit County. The deposit is about two miles southeast of Robinson, and about a mile and a quarter east of Climax, stations on the Colorado & Southern Railway. Its location is shown in Plate XIV. At the time of the writer's visit to the property in the late fall of 1913 it was covered with snow and little first-hand information concerning the deposit could be obtained. In places where the snow had blown off large quantities of slide rock were exposed, which on an average appeared to contain 0.5 to 1 per cent molybdenite, the mineral occurring in the cracks and fractures of a crystalline quartz, most of which was associated with more or less pyrite, but was practically free from copper minerals. Although most of the deposit was covered with snow, the author had no difficulty in picking up from this particular slide within a few minutes 100 pounds of rock which on sampling and analysis yielded 1.06 per cent MoS_2. A specimen of typical ore is shown in Plate XII, *B*.

Eight adjoining claims are reported to have been located on the deposit. The chief development work at the time of the writer's visit consisted of two tunnels, one near the northwestern end of the deposit, on the claims of H. Leal, and the other near the center of the deposit, on the Gillaspey claim, known as "Denver No. 2." The first tunnel had been driven in an eastern direction for a distance of over 500 feet, and was being pushed ahead with the idea of crosscutting the deposit. As far as work had progressed the character of the rock seemed essentially the same, and the writer was told that samples taken at frequent intervals throughout the entire length of the tunnel showed an average content of 0.7 per cent MoS_2. The second tunnel was said to be 30 to 40 feet long, but as its portal was closed by a snowdrift it was not entered.

The following notes concerning recent development on the deposit were kindly furnished the writer by Ira Snyder, of Denver, Colo., who has purchased the "Denver No. 2" claim:

> The tunnel on the Leal claims has been extended to a total length of 800 feet and at the face a 100-foot drift runs at right angles to the line of the tunnel. No walls have been encountered and there has been no perceptible change in the character or average richness of the ore. A lease is said to have been taken on the Leal claims by Messrs. McDonald, Noble, and King, of Leadville, who have built a wagon road to the property from Climax. These men are also reported to have mined several hundred tons of ore from the claims of Charles J. Senter, near the southeastern end of the deposit, shipping the same to Leadville for concentration by flotation in an experimental plant. The tunnel on the "Denver No. 2" is stated to be now about 75 feet in length, and the ore encountered is reported to contain an average of about 1.3 per cent MoS_2. Experiments on a large scale are being carried on in Denver for concentrating by flotation the ore from this claim. The results of these tests have been satisfactory, and preparations are being made for the installation of a plant at the mine during the coming summer.

MOLYBDENITE NEAR OPHIR, SAN MIGUEL COUNTY.

The Molybdenite Queen claim, owned by G. C. Knox, of Denver, and J. Belisle, of Norwood, Colo., is situated in the Iron Spring mining district about 1 mile southeast of the town of Ophir, which is 2½ miles east of Ophir Loop, a station on the main line of the Rio Grande Southern Railroad. It crosses Nevada Gulch about 600 feet above the creek at the base of the hill and adjoins the Favorite group of gold claims. The deposit is said to consist of a well-defined vein of white quartz 20 to 25 feet wide, traversing a poryphyry country rock. The molybdenite occurs in the vein in flaky crystal aggregates and irregular-shaped masses from the size of a pea to pieces three-quarters of an inch or more in diameter. In a 25-pound lot of ore, said to be a sample taken on the surface across the width of the vein, the writer would estimate the molybdenum sulphide content to be 2 to 3 per cent. In this sample considerable alteration of the molybdenite to molybdite had taken place and a small quantity of pyrite was noticed. The ore appears of such character as to be readily concentrated by either electrostatic or flotation methods.

The vein is said to strike in a general southwestern direction, but its dip has as yet not been determined. Practically no development work has been done on the outcrop, but the vein is reported to be cut at a depth of about 600 feet by the Deadwood tunnel and to be approximately 20 feet wide at the place of its intersection. However, the owners of the property have been unable to verify this statement, as entrance to the tunnel can not be had, owing to a cave-in only a few feet from the portal.[a]

OTHER OCCURRENCES OF MOLYBDENITE IN COLORADO.

DEPOSIT NEAR ROBINSON, SUMMIT COUNTY.

A considerable deposit of low-grade molybdenite ore, practically identical with that of Bartlett Mountain described above, is situated on Chalk Mountain near Robinson, Summit County. (See Pl. XIV.) Four claims located on the deposit are owned by E. G. Heckendorf, J. W. Harris, and S. H. Weber, of Denver, and J. A. Weber, of Leadville, Colo. The ore body is reported to be 30 to 40 feet wide, and its outcrop, which runs northeast, is said to have been traced for over 3,000 feet. In October, 1915, development work consisted of seven open cuts, samples from which are reported to have assayed 1 to 3 per cent of molybdenum disulphide.

DEPOSIT NEAR NATHROP, CHAFFEE COUNTY.

Bert Bergstrom and H. C. Hayes, of Buena Vista, Colo., are said to have opened a promising molybdenite property in Chaffee County, a few miles south of Fisher, a station on a branch of the Colorado & Southern Railroad from Nathrop. It is understood that the molybdenite occurs in quartz in flakes up to an inch in maximum diameter and that the ore is practically free from copper minerals. About 1 ton of high-grade molybdenite is reported to have been obtained by careful cobbing and hand sorting of some of the richer ore. It is said that development work on the property is going ahead rapidly.

PROSPECT NEAR PARKDALE, FREMONT COUNTY.

Ralph Fairchild, of Canon City, Colo., has sent the Bureau of Mines specimens of molybdenite ore that is said to occur in considerable quantity in a prospect being developed for copper 7 miles south of Parkdale, Fremont County. The specimens show 5 to 6 per cent of molybdenite associated with pyrite and chalcopyrite in a siliceous gangue. Development work on the property consists of a shaft about 100 feet deep.

An occurrence of molybdenite is reported in Fremont County on the northern slope of Rito Alto Peak in the Sangre de Christo Range. The deposit is above timber line, at an altitude of over 11,000 feet, and is difficult of access, being 18 or 20 miles from Cotapaxi, the nearest station on the Denver & Rio Grande Railroad, and the last 6 or 8 miles of the journey to the property is over a very rough trail. It is understood that the only development work consists of a shaft about 30 feet deep. Specimens of ore said to have come from this occurrence and sent to the writer by B. J. Hillman, of Denver, Colo., consist of a quartzose rock in which the molybdenite occurs as flakes and irregular-shaped masses up to one-half inch or more in diameter and constitutes perhaps 4 or 5 per cent of the whole. The molybdenite is associated with chalcopyrite. No information regarding the size of the deposit nor the general grade of the ore is available.

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[a] The above information is from a report given the writer by G. C. Knox, of Denver, Colo., on Nov. 8, 1915.

SPECIMENS FROM WESTCLIFFE, CUSTER COUNTY.

Specimens of molybdenite ore said to be representative of a considerable quantity of material on the dump of an old mine 12 miles north of Westcliffe, Custer County, have been submitted to the bureau by L. H. Schoolfield, of that town. The rock in these specimens is so altered that its identification is impossible. It appears to be a highly metamorphosed calcareous sediment, and, besides the molybdenite, which occurs in flakes up to one-fourth inch in maximum diameter, it contains siderite, calcite, quartz, and some feldspar. It is seemingly free from copper. The writer estimates the molybdenite content of the samples submitted as 2 or 3 per cent.

CLAIM NEAR MARBLE, GUNNISON COUNTY.

The Bureau of Mines has received two samples of molybdenite ore said to have come from a claim recently located by John O'Toole, of Denver, on the southwestern slope of Treasury Mountain, about 8 miles north of Marble, Gunnison County. One sample consists of milky quartz, in which the molybdenite, associated with considerable molybdite, occurs in flakes up to one-quarter of an inch in diameter. In the other sample the quartz is crystalline and the molybdenite more granular in character. Both lots of ore were free from copper minerals and appeared to contain 2 to 3 per cent of molybdenum disulphide. The ore was said to have been taken from two roughly parallel veins about 150 feet apart and striking northeast. The average width of the larger vein was stated to be about 3 feet and that of the smaller about 20 inches.

DEPOSIT SOUTH OF ASPEN, PITKIN COUNTY.

Molybdenite is reported to occur in a deposit owned by F. E. Kendrick and associates, of Leadville, Colo., and situated south of Red Mountain about 20 miles south of Aspen, Pitkin County. The vein, which is called the Green Horn lode, is said to be 6 feet wide. A specimen of ore purported to have come from this deposit consists of a brecciated quartz heavily impregnated with pyrite and containing occasional fine flakes of molybdenite.

CLAIMS NEAR KOKOMO, SUMMIT COUNTY.

G. M. Bacom, of St. Elmo, Colo., has submitted to the bureau samples of molybdenite ore said to have come from a claim near Kokomo, Summit County. The ore consists of white crystalline quartz containing a small amount of fine-grained molybdenite and its alteration product, molybdite. It seems identical with the molybdenite ores from Bartlett and Chalk Mountains which are described above. No information regarding the exact location or size of the deposit is available.

Small quantities of flaky molybdenite occur in ore from the tunnel on claims belonging to Joseph Bryant, of Kokomo, Summit County, and situated about 2 miles southeast of that town. The ore consists principally of quartz heavily impregnated with pyrite.

PROPERTY NEAR ST. CLOUD, LARIMER COUNTY.

E. F. Bartlett, of St. Cloud, Larimer County, has submitted specimens of magnetite containing occasional patches of molybdenite. This ore is said to have come from a property about 4 miles south of the town.

MONTANA.

Out of a dozen or more authenticated occurrences of molybdenum ore in Montana, only two occurrences of molybdenite and one of wulfenite have been examined by the writer. Unfortunately, these deposits were of necessity visited during the late fall when they were covered with snow, which precluded an examination of their outcrops and otherwise hampered their investigation. Besides the occurrences of molybdenite listed in the accompanying table, two occurrences of wulfenite have been reported in the State. One of them is at Radersburg,[a] in Broadwater County, and the other is near Twin Bridges, in Madison County. The latter deposit was visited and is described hereafter.

Occurrences of molybdenite in Montana.

Location.	Remarks.	Reference.
Beaverhead County:		
Head of Birch Creek on Tent Mountain, 12 miles west of Apex and 30 miles northwest of Dillon.	In quartz	Mineral Ind., vol. 13, 1904, p. 328, Mineral Resources U. S., 1903, U. S. Geol. Survey, 1904, p. 308, and Mineral Resources, U. S., 1904, U. S. Geol. Survey, 1905, p. 341.
Wise River	See p. 76	Letter from F. W. Hall, Wise River, Mont.
Broadwater County:		
At Winton	With calcite and vesuvianite	Specimen in U. S. National Museum.
Fergus County:		
At Neihart		U. S. Geol. Survey Bull. 585, 1914, p. 112.

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[a] Specimen in Harvard collection, Agassiz Museum.

Occurrences of molybdenite in Montana—Continued.

Location.	Remarks.	Reference.
Jefferson County:		
At Clancey	With feldspar, quartz, chalcopyrite, and muscovite.	Specimen in U. S. National Museum.
Near Homestake		Mineral Resources U. S., 1906, U. S. Geol. Survey, 1907, p. 528; Eng. and Min. Jour., vol. 76, 1903, pp. 370, 791.
Madison County:		
Near Sheridan		Mineral Resources U. S., 1906, U. S. Geol. Survey, 1907, p. 528.
Missoula County:		
Leslie Copper Mine in western part of county.	In quantity on 150-foot level	Mineral Resources U. S., 1901, U. S. Geol. Survey, 1902, p. 266.
Park County:		
Near Chico Hot Springs	See p. 73	
Near Cooke, at head of Stillwater River.	See p. 76	
Powell County:		
In Carpenter Gulch, 2 miles east of Ophir.	See p. 75	Mineral Resources U. S., 1903, U. S. Geol. Survey., 1904, p, 308, and Mineral Resources U. S., 1904, U. S. Geol. Survey. 1905, p. 341.
Silverbow County:		
In the Montgomery, Altoona, and neighboring claims and number of places east of the Flat and in the Gagnon mine.		Weed, W. H., Geology and ore deposits of the Butte district, Montana: U. S. Geol. Survey Prof. Paper 74, 1912, p. 79.

MOLYBDENITE NEAR CHICO HOT SPRINGS, PARK COUNTY.

The molybdenite property of the Great Western Mining & Milling Co. is situated in Park County about 6½ miles from Chico Hot Springs and 5½ miles southeast of Chico. It is at an elevation of about 7,500 feet on the northeast bank of Emigrant Creek opposite Emigrant Peak, and on the southwest slope of the ridge joining Chico Peak and Mineral Mountain, outlying peaks of the Absaroka Range.

The property is about 10 miles from Emigrant, the nearest railroad point on the Yellowstone Park branch of the Northern Pacific Railroad from which it is reached by wagon road via Chico Hot Springs and Chico. From Emigrant to Chico this road is in fair condition and there are no bad grades, but up Emigrant Creek the road rises almost continuously and many of the grades are so steep that the hauling of even moderate loads is accomplished with difficulty.

The deposit consists of a vein of what is seemingly a brecciated and somewhat altered fine-grained light-gray porphyry in which fine-grained molybdenite intimately associated with pyrite forms the matrix and permeates the individual fragments of brecciated material. Plate XIII, *B*, illustrates a cross section of typical ore from the deposit. According to Iddings and Weed[a] the rocks downstream from the mine consists of basic andesitic breccia and flows, whereas above the mine they are acid porphyrites, andesite, and dacite. At the time the writer visited the property, in the late fall of 1913, the outcrop of the vein was covered with snow and the underground workings were inaccessible owing to the presence of a great quantity of carbonic acid gas. As the writer was unable to make an examination of the workings, H. F. Lawrence, the mine manager, kindly supplied the following notes:

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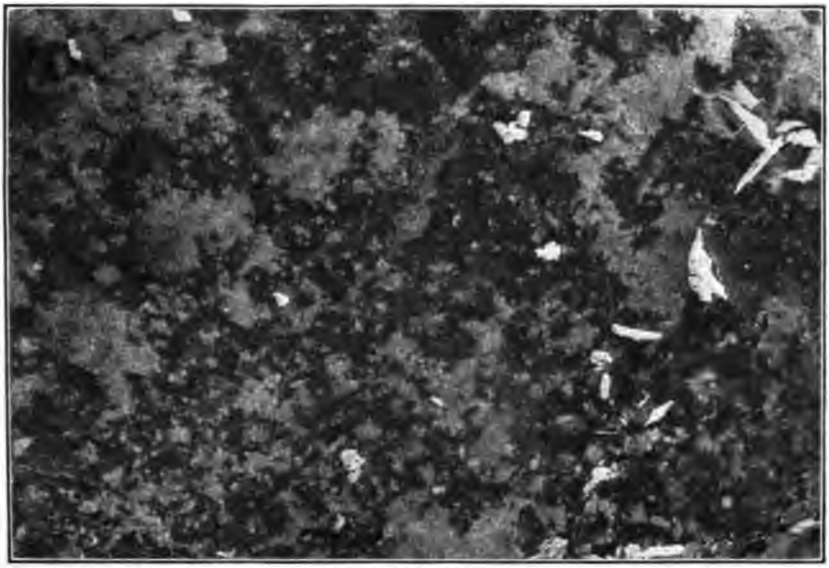
[a] Iddings, J. P., and Weed, W. H., Livingston folio (No. 1), Geol. Atlas U. S., U. S. Geol. Survey, 1894.

> The molybdenite deposit was cut many years ago by a tunnel driven in search of gold. This old tunnel (said to be 700 to 800 feet long but the face of which is inaccessible owing to cave-ins) intersects the molybdenite-bearing vein 360 feet from the portal and at a vertical depth of about 260 feet below the outcrop of the vein. At the point of intersection by the tunnel the vein is about 14 feet wide and so nearly perpendicular that it is impossible to say which is the foot and which the hanging wall. Only one wall is well defined. There is practically no development work on the vein at the tunnel level, only a few feet of drifting having been done and a raise started. The strike of the vein is approximately N. 30° W. by S. 30° E. It has been traced on the surface for over 1,000 feet by means of open cuts, and float ore indicates that it continues much farther. The average width of the outcrop is 12 to 15 feet.

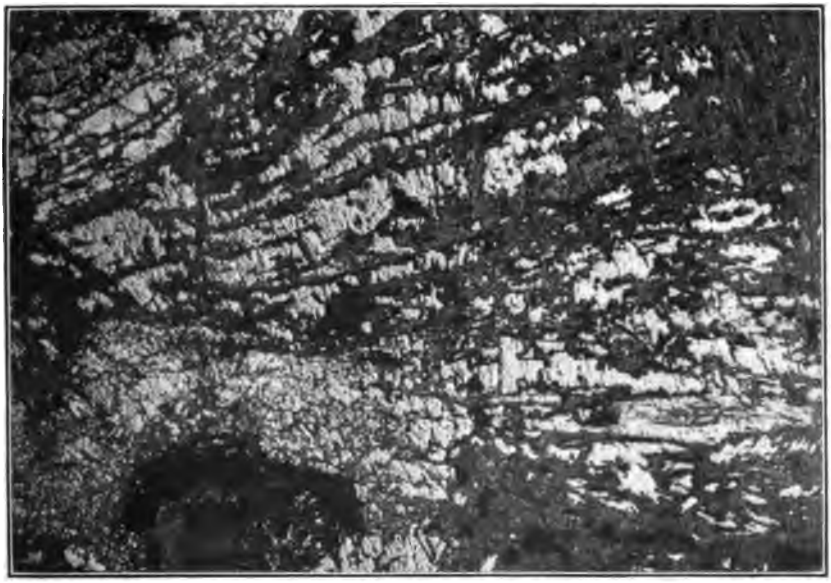
As regards the molybdenite content of the ore, the author was informed by E. C. Sackett of Livingston, Mont., that two samples taken by him across the drift in the tunnel assayed 14 and 23 per cent MoS_2, respectively, but these samples were probably taken in the richest places that could be found. As indicated by several tons of ore in a small mill erected for experimental concentration work near the mouth of the tunnel, the ore in the drift is of good grade, averaging perhaps 5 to 6 per cent MoS_2. As far as shown by tests in a few small specimens, the ore contains no copper.

At the time of the writer's visit 10 to 12 men were employed in gathering a fuel supply for winter. A new boarding house had just been completed and the foundation for a mill erected, and active development work was contemplated within a few weeks. It was proposed to treat the ore by water flotation without the use of acid or oil on machines designed by George B. Allison, president of the company. Whether the proposed process of treatment will be successful can not be foretold, but from a number of concentration tests on a model machine, which the writer saw made at Butte, Mont., it appears to be impossible to obtain a high-grade concentrate (85 per cent or more MoS_2) from the ore by the process contemplated. The association of the molybdenite with the gangue is intimate and it is stated that the ore must be crushed through a 60-mesh to 80-mesh screen before the molybdenite is liberated. It is practically impossible to feed this fine material to the water surface in an ore sheet only one mineral particle in depth, and for this reason considerable fine silica is supported on the floating film of molybdenite. A part of the pyrite in the ore also floats, and both of these impurities detract considerably from the grade of the concentrates.

Plenty of wood for both fuel and mine timbers can be obtained in the immediate vicinity of the mine, and Emigrant Creek carries sufficient water to supply a large mill.



A. SECTION THROUGH MOLYBDENITE ORE FROM QUIGLEY MINE, CARPENTER GULCH, NEAR OPHIR, POWELL COUNTY, MONT. (SLIGHTLY ENLARGED.)



B. SECTION THROUGH MOLYBDENITE ORE FROM ROMERO MINE, PORVENIR, SAN MIGUEL COUNTY, N. MEX. (TWO TIMES NATURAL SIZE.)

Inquiries addressed to the president of the company as to the progress made in development work have not been answered; hence the author can not state what the result of this work was or whether it was ever carried out as contemplated.

MOLYBDENITE IN CARPENTER GULCH, POWELL COUNTY.

There is an occurrence of molybdenite ore about 2 miles east of Ophir, in Carpenter Gulch, Powell County. The property, which is owned by Thomas L. Quigley, of Sacramento, Cal., is about 7½ miles from Avon and 12 miles from Elliston, stations on the Northern Pacific Railroad, from which it is reached by wagon road. The elevation of the mine is approximately 5,700 feet. The author visited the property in the late fall of 1913, when the outcrop of the deposit was covered with snow. Apparently the property had not been worked for several years, and the portal of the tunnel by which it was developed was locked. Furthermore, the tunnel had caved badly near the portal, so that access to it could not be had. By clearing away the snow from a part of the dump the writer was able to obtain 30 or 40 pounds of ore, containing perhaps 2 or 3 per cent of molybdenite in flakes and irregular-shaped masses up to an inch in maximum diameter. The rock in which the molybdenite occurs is seemingly a metamorphosed limestone that has undergone various stages of alteration. Some specimens are much more highly altered than others. The great differences in the character of individual specimens of the ore are illustrated by Plate XVI, *A*, which also plainly shows how the molybdenite occurs. The most prominent minerals in the rock are garnet, epidote, quartz, some unidentified silicates, and one or more calcareous carbonates. The ore is seemingly free from copper minerals.

Tests of the crushed ore show that the garnet contained in it is a good conductor of electricity, and therefore concentration by electrostatic methods is impracticable, but the ore is evidently suitable for treatment by flotation. From the size of the dump at the tunnel mouth the writer would estimate that the tunnel had been projected between 150 and 200 feet, but as the tunnel was inaccessible for the reasons stated above nothing definite can be said here as to the size of the deposit and of the possibilities of its being of commercial importance.

A second occurrence of molybdenite in Carpenter Gulch is reported at the head waters of the creek, about 2½ miles above the occurrence already noted.[a] Specimens said to have come from this deposit show the molybdenite occurring in small flakes in what is seemingly a metamorphosed limestone.

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[a] Letter, dated Mar. 6, 1915, from C. L. Smith, Deer Lodge, Mont.

OTHER MOLYBDENITE OCCURRENCES IN MONTANA.

The Bureau of Mines has received from G. H. Gibson of Cooke, Mont., several samples of molybdenite ore said to have come from a deposit on the headwaters of the Stillwater River near Cooke, Park County. The samples consist of greenish-black, medium to coarse grained garnetiferous amphibolite in which the molybdenite occurs in small irregularly distributed patches. The rock is heavy and tough, and at least 75 per cent of it is made up of dark-green hornblende. The other principal constituents are a reddish-brown garnet and quartz, the latter occurring for the most part in narrow veinlets. Pyrite occurs associated with the molybdenite both in the hornblende and in the garnet. In the 30 or 40 pounds of material submitted to the bureau the author would estimate the molybdenite content to be 1 to 1.5 per cent.

B. R. Holland of Cooke, Mont., has sent in specimens of quartz containing small flakes of molybdenite associated with molybdite. These are said to have come from a prospect in the vicinity of that town.

Specimens of molybdenite ore, said to have been obtained from stringers and small veins cut in driving a tunnel in Comet Mountain on the property of the Boston-Montana Development Co., Beaverhead County, have been received from F. W. Hall, of Wise River, Mont. The molybdenite occurs as small flakes in quartz and is stated to occur in quantity.[a]

WULFENITE ORE NEAR TWIN BRIDGES, MADISON COUNTY.

The property of the Grand View Mining & Development Co., of Twin Bridges, Mont., is situated near the crest of the Tobacco Root Range, about 11 miles east of Twin Bridges, the nearest station on a branch of the Northern Pacific Railroad from Whitehall. It is situated near the summit of Little Baldy Mountain and is reached by wagon road up Goodrich Gulch from Twin Bridges.

The deposit consists of a vein of porous and well-crystallized cerussite heavily impregnated with iron oxide and containing perhaps ½ to 1 per cent of well-crystallized wulfenite. The ore is reported to carry 30 to 50 per cent of lead, 2 to 6 ounces of silver, and 0.2 ounce to 1.5 ounces of gold per ton.

At the time of the author's visit to the property in the late fall of 1913 little development work had been done, and the outcrop of the deposit was covered with several feet of snow, so little exact information as to the size and extent of the deposit could be obtained. A short tunnel had been driven only a few feet below the outcrop, and intersected the vein at a point where it was 17 feet horizontally between walls, and a few feet of drifting had been done on the vein at this point. The strike of the vein as indicated by this work was practically east, and the dip appeared to the author to be about 35° to 40° to the north. However, the management of the property seemingly has a radically different idea as to the dip of the deposit, because the company has started a tunnel several hundred feet below, on the south side of the mountain, and is driving it north to intersect the vein, whereas if the deposit dips in the direction the author supposes the vein could be much more readily reached by a tunnel from the north side of the mountain. In the upper workings black limestone seems to form both the footwall and the hanging wall of the deposit.

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[a] Letter of Dec. 9, 1914, from F. W. Hall, Wise River, Mont.

A microscopic examination of a large dark-colored wulfenite crystal, said to have come from this deposit and given to the author by Henry Schmidt, president of the company, showed the presence of metallic gold in the crystal, the occurrence being seemingly similar to that of the native metal in wulfenite crystals from the Old Yuma mine near Tucson, Ariz. (see p. 115).

Because of the small amount of development work on the property, it is impossible to say whether the occurrence of wulfenite in the ore may prove of commercial importance as a source of molybdenum. However, if concentration of the ore is attempted with a view to saving the wulfenite, great difficulty is almost certain to be encountered in separating it from the cerussite, which is nearly of the same specific gravity.

NEW MEXICO.

Few occurrences of molybdenite have been reported in New Mexico, and only one deposit in the State, that of the Romero Mining Co., near Porvenir, San Miguel County, has been visited by the author. Other occurrences are at Rociada, about 6 miles north of Porvenir, in the same county; at Bromide, in Rio Arriba County; and at the Modoc mine, in the Fierro district, Grant County. In all these localities the molybdenite is associated with copper ores.[a]

The mineral also occurs in quartz breccia in a deposit near Magdalena, Socorro County,[b] and associated with lead and silver ores at Organ, Dona Ana County.[c]

Deposits of wulfenite are confined almost entirely to Dona Ana, Sierra, and Grant Counties, in the southwestern corner of the State. The mineral occurs rather commonly with silver ores in the Organ district, in Dona Ana County, and many well-crystallized specimens have been obtained from the Bennett mine at Organ. Most of these specimens show beautiful yellow crystals of wulfenite associated with cellular quartz and secondary calcite and limonite. In Grant County the mineral occurs in deposits near Lordsburg[a] and also in the Lucky Bill mine near Santa Rita, but as far as known these occurrences are only of mineralogical interest. Plate IV, *B*, shows a group of wulfenite crystals of unusual form from the Lucky Bill mine. In Sierra County wulfenite has been mined at Hillsboro,[b] and a small production of the mineral was derived by Mr. Ralph Widener in 1914 from the Gladys claim, in the Caballos Mountains,[c] near Cutter. The occurrence of the mineral has also been reported from the Belcher mine, in Santa Fe County.[d]

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[a] Sanford, Samuel, and Stone, R. W., Useful minerals of the United States: U. S. Geol. Survey Bull. 585, 1914, p. 132.

[b] Specimens in collection of Bureau of Mines.

[c] Sanford, Samuel, and Stone, R. W., loc. cit.

MOLYBDENITE PROPERTY OF THE ROMERO MINING CO.

The Romero Mining Co., of Las Vegas, has done considerable development work on a molybdenite prospect situated about a mile and a half from the Porvenir Hotel at Porvenir, San Miguel County. This town is about 18 miles northwest of Las Vegas, and may be reached from there over a good wagon road or over a part of the same road from the northerly terminal of the Hot Springs Railroad, from which it is about 8 miles distant.

The vein material of this deposit is seemingly a pegmatite, consisting largely of quartz and feldspar, with smaller amounts of biotite and occasionally fluorite. The molybdenite, which is closely associated with chalcopyrite, occurs in masses up to 2 or 3 inches in maximum diameter in occasional pockets in the vein material. The ore also contains scheelite, bismuthinite, malachite, molybdite, and various iron oxides. Plate XVI, *B*, illustrates a cross section of a specimen of molybdenite ore from this property. The interesting ore structure shown, although not typical of the ore as a whole, was noted in a number of specimens. The country rock is a fine-grained pink feldspathic granite, which upon microscopic examination shows the following minerals: Quartz, orthoclase, microcline (very abundant), albite, a small amount of biotite, and a few minor accessory minerals.

When the author visited this property, in the winter of 1913, it was covered with snow and the outcrop of the deposit could not be examined. At that time development work consisted of a tunnel about 700 feet long and a shaft said to be about 75 feet deep. The bottom of this shaft was filled for 20 feet or more with material that had caved from the sides. Twenty or thirty tons of rich molybdenite ore associated with tungsten, bismuth, and copper minerals was reported to have been taken from several pockets in this shaft, but at the time of the author's visit there was no ore visible either in the shaft or in the tunnel, which was being driven with the expectation of crosscutting the ore-bearing pegmatite at depth. A considerable part of the rich ore taken from the shaft was sacked and stored in a small experimental mill that had been erected near the property. A part of it had been treated by crushing, followed by screening and hand picking to remove the coarser pieces of molybdenite, followed by concentration on a Wilfley table. The author was shown several small lots of coarse high-grade molybdenite and several tons of concentrates containing about 7 per cent MoS_2 and rich in copper, which was obtained by this method of treatment. It was stated that tests to recover the molybdenite and copper by means of an oil-flotation process tried on an experimental scale at the mill had resulted in failure. It is understood that a few hundred pounds of coarse high-grade molybdenite, which presumably was recovered from some of the sacked ore in storage at the time of the author's visit, was sold by the company in 1914.

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[a] Sanford, Samuel, and Stone, R. W., op. cit., p. 134.

[b] Sanford, Samuel, and Stone, R. W., loc. cit.

[c] Hess, Frank L., The production of cobalt, molybdenum, nickel, etc.: Mineral Resouces of U. S. for 1914, U. S. Geol. Survey, 1915, p. 925.

[d] Specimen 84293 in U. S. National Museum.

WASHINGTON.

CROWN POINT MINE.

The Crown Point mine of the Aurelia Crown Co. of Seattle is situated in the Chelan National Forest, in the NW. ¼ T. 31 N., R. 16 E., at the head of Railroad Creek, Chelan County. It is near the summit of the Cascade Range at an elevation of over 5,000 feet. (See accompanying sketch map, fig. 1.) The property is reached from Chelan Falls, on the Great Northern Railway, via stage to Chelan or Lakeside, a distance of 3 or 4 miles, thence by steamer up Lake Chelan for about 40 miles to Lucerne, and then by wagon road and trail up Railroad Creek for about 18 miles. The first 3 or 4 miles of this last part of the journey are over a poor wagon road which ascends the hill back of Lucerne with many steep grades and sharp curves to connect with an abandoned railroad grade which continues up the north bank of the creek to a point about 12 miles from its mouth. From the up-stream end of the railroad grade a poor trail leads to the property, about 6 miles distant. The elevation of Lake Chelan at Lucerne is 1,072 feet. Accordingly the vertical rise from the lake to the mine is about 4,000 feet, and the average grade of the road and trail for the entire 18 miles is over 4 per cent.

The molybdenite-bearing deposit at the Crown Point mine consists in the main of a nearly horizontal vein of white vitreous quartz, which outcrops for a distance of several hundred feet on the northeast face of an almost perpendicular granite cliff at a height of 700 or 800 feet above its base. This vein dips slightly (perhaps 5° to 6°) west. On the outcrop its maximum thickness is about 3 feet, and it pinches gradually to 3 or 4 inches at both ends of the outcrop. The vein also decreases rapidly in thickness as it runs back into the cliff, and in a distance of 75 to 100 feet it practically pinches out. Plate XVII, *A*, shows the outcrop at the mouth of one of the short tunnels by which the property is developed. Attention is called to the pinch of the vein toward the left as it runs into the cliff and to the well-defined footwall and hanging wall. The old tree stub at the right leans out from the face of the cliff, which drops almost perpendicularly from the mouth of the tunnel for 600 or 700 feet.

An inspection of the local topography makes it evident that the hill in which the vein occurs once extended much farther to the northwest, and that a considerable part of it has been removed by erosion to form the valley on which the present cliff faces. It is certain that the molybdenite-bearing vein extended into this eroded part, but there is no evidence from which to determine how much of the vein has been carried away.

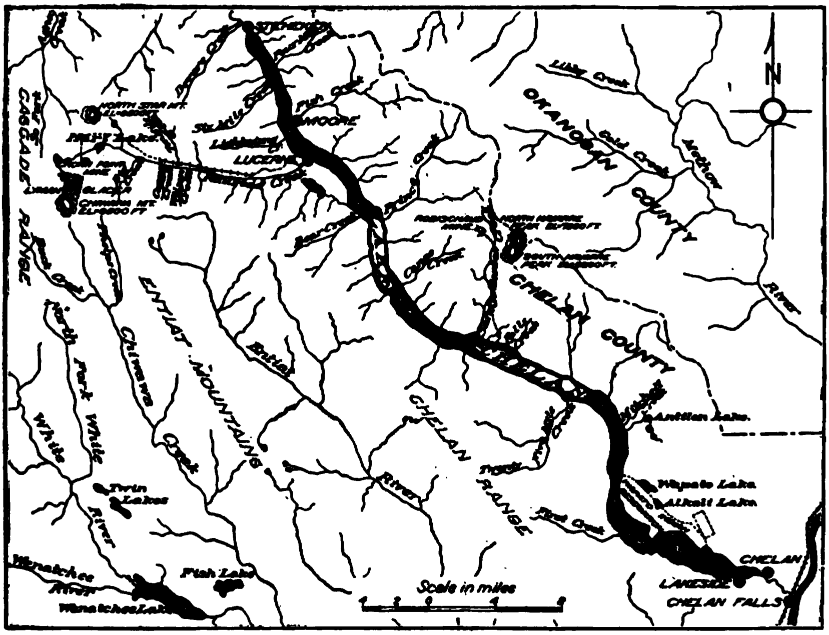
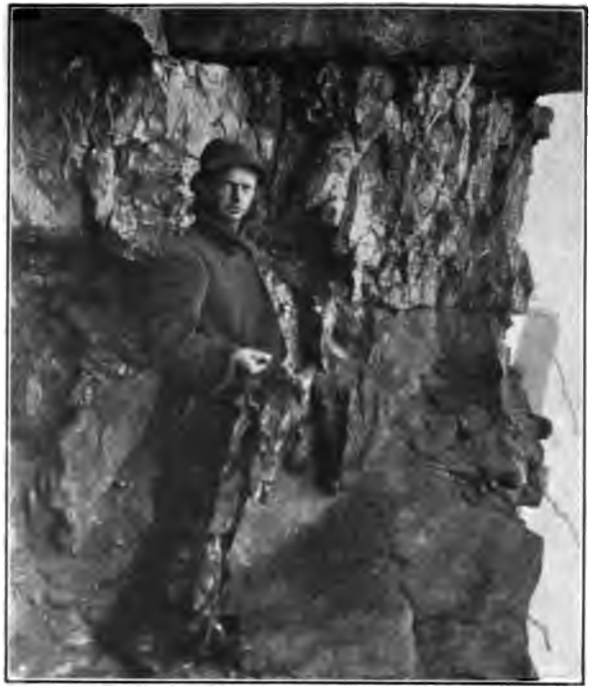
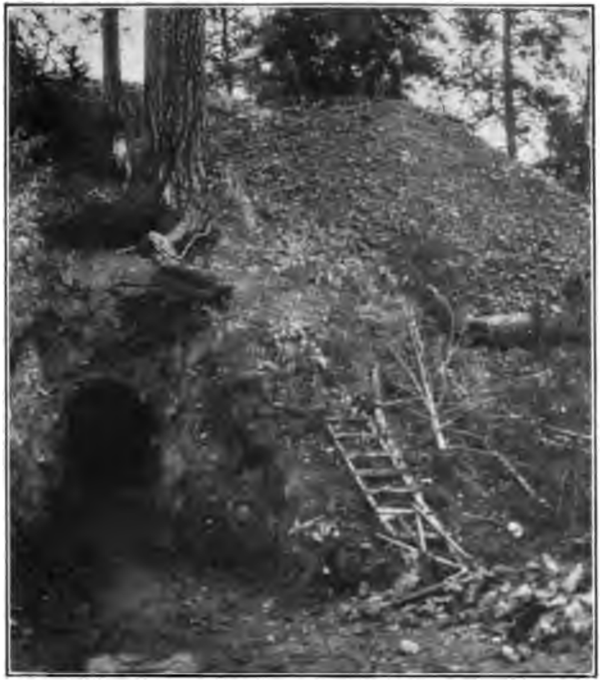


FIGURE 1.—Sketch map of the vicinity of Lake Chelan, Wash., and vicinity, showing the situation of the Crown Point and Robischand mines.

The molybdenite occurs scattered irregularly through the quartz in masses ranging in size from minute specks to pieces 4 to 5 inches in maximum diameter. In general the molybdenite is unusually well crystallized, and the deposit probably affords finer specimens of the mineral than are to be obtained from any other known occurrence. Most of the crystals have the form of flat, hexagonal pyramids, the majority of which are beautifully striated. Some of them are one-half to 1 inch high, and 4 to 5 inches in maximum diameter, and weigh one-fourth to one-third of a pound each The contrast afforded



A. OUTCROP OF UPPER VEIN AT MOUTH OF TUNNEL NO. 1, CROWN POINT MINE, RAILROAD CREEK, CHELAN COUNTY, WASH.



B. LOWER TUNNEL AND DUMP FROM UPPER WORKINGS, STARR MINE, NEAR LOOMIS, OKANOGAN COUNTY, WASH.

by these lustrous black crystals on a background of clean white quartz is strikingly beautiful. Photographs of some of these crystals and of a characteristic ore from this deposit are reproduced in Plates I and III, *A*. A few masses of molybdenite larger than those mentioned above are found, and the company states that "some have been mined that weighed over 5 pounds," and that "one weighed 11 pounds." In general, the molybdenite does not occur in the center of the vein, but is confined to horizons extending from a line 2 to 3 inches from the center of the vein to a line the same distance of either wall. The accessory minerals are unimportant. Small quantities of chalcopyrite and copper carbonates derived from it occur at several places in the vein, also occasional small patches of galena and sphalerite. These minerals are, however, largely confined to occurrences along the walls or in the immediately adjoining country rock. On the whole, the quartz-vein material is remarkably free from all minerals except molybdenite. In several places along the outcrop and in the tunnels where molybdenite is exposed to the air it has altered to molybdite, and fine specimens of partly altered crystals may be obtained.

The country rock is a medium to fine grained, greenish-gray biotite granite in which the feldspars are kaolinized to a considerable extent. The biotite is also more or less altered and is in various stages of chloritization. According to analyses made by Zaumbrecker and Pierce,[a] of Northwestern University, the country rock does not contain molybdenum. Crook, in describing the occurrence of molybdenite at this mine,[b] cites the absence of molybdenum in the neighboring granite as an indication that the molybdenite in the vein has not been derived by lateral secretion.

About 10 feet immediately below the vein already described and roughly parallel with it there is a second smaller quartz vein which outcrops for a short distance a long the face of the cliff. The vein is only a few inches in thickness, but it is said to be richer in molybdenite than the larger vein. Little development work has been done on it, and nothing can be said of its possibilities.

Development work on the upper vein consists of two short intersecting tunnels. The larger of these, locally known as Tunnel No. 1, runs about 200 feet west and is intersected at an acute angle by Tunnel No. 4, which is about 80 feet long and runs southwest. The author's examination of the property was of necessity confined almost entirely to an inspection of the upper vein as disclosed by these two tunnels, as a heavy snowstorm, in progress at the time of his visit, rendered work along the outcrop on the face of the cliff impossible.

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[a] Crook, A. R., Molybdenite at Crown Point, Washington: Geol. Soc. of America Bull., vol. 15, 1904, p. 287.

[b] Crook, A. R., op. cit., pp. 283-288.

Equipment to serve the Crown Point and neighboring claims controlled by the same owners consists of a mill, machine shop, power house, sawmill, warehouse, three cabins, and a barn. The mill is situated on the slope at the foot of the cliff in which the molybdenite-bearing veins occur, and is connected with the tunnels above by an aerial tram. As the property had been closed for the winter at the time of the author's visit, entrance to the mill was not to be had, and no detailed description of its contents can be given here. The equipment is said to consist of crushing and concentrating machinery, the latter including a Schule dry concentrator imported from Hamburg, Germany, for treating the molybdenite ore.[a] The mill building also contains a small compressor plant operated by a Pelton wheel. The president and general manager of the company in speaking of the mill stated: "All the machinery works well except the Schule concentrator, which does not save the small particles of molybdenite; neither does it entirely separate all the quartz from the molybdenite we have concentrated."

There is a splendid supply of timber available for use in mining or building. About half of this is Douglas and white fir and the remainder consists of cedar, white and yellow pine, spruce, and hemlock. As already stated, the property lies within the boundaries of the Chelan National Forest: hence stumpage is for sale by the United States Government.

Much water power could be developed from a number of sources in the immediate neighborhood. Principal among these is Aurelia Lake, which has an area of about 80 acres, and is said to be capable of supplying water with a head of more than 1,600 feet within the confines of the property.

The mine is credited with a production of 10 to 12 tons of molybdenite ore in both 1901 and 1902.[b] Presumably this was high-grade material, although no statement regarding its quality is made. After leaving the property the author saw at the warehouse of the company at Lucerne about 2½ tons of high-grade mineral estimated to contain over 95 per cent MoS_2 which was ready for shipment. This material was said to have been obtained by hand cobbing the ore and picking out the larger pieces of molybdenite.

The cost of mining at the property is considerable on account of the high cost of transporting supplies and the narrowness and slight dip of the ore body. The granite in which the vein occurs is hard and at least 3 or 4 feet of it must be mined. However, the vein stands up fairly well after the underlying granite has been removed, and with careful selective mining little sorting would be necessary, and the little necessary would be rendered easy on account of the great dissimilarity between the vein material and the waste rock.

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[a] Editorial, Eng. and Min. Jour., vol. 78, 1904, p. 273.

[b] Pratt, J. H., Tungsten, molybdenum, uranium, and vanadium: Mineral Resources U. S. for 1902, U. S. Geol. Survey, 1903, p. 287.

The precipitation during both summer and winter is great, and, owing to the altitude of the property, snow remains until early summer. The roads and trails from Lake Chelan to the property are generally open in June, and easy access to the mine may be had until the middle or last of September. On September 15, 1914, the company's men, who were carrying on development and assessment work, left the property for the winter, and on the next day, when the author visited the property, a heavy fall of snow took place.

If further development should disclose sufficient ore of similar character to that already in sight to warrant the installation of the necessary machinery, no difficulty should be experienced in concentrating the ore. A large part of the molybdenite could be efficiently recovered by hand picking after crushing the coarse pieces. The discard might then be reduced to perhaps 10 mesh and treated by electrostatic methods. Concentration tests of nearly similar ores lead the author to believe that a high recovery and a high-grade concentrate could be made.

MOLYBDENITE NEAR LOOMIS, OKANOGAN COUNTY.

There is a large deposit of low-grade molybdenite ore about 10 miles from Tonasket, Okanogan County, on one of the spurs of the Aeneas Mountains. It may be readily reached by automobile from Tonasket or from Loomis, both towns being about the same distance from the property. The deposit, which is owned by Andrew Starr, of Tonasket, appears to consist of a blow-out or chimney of medium-grained light-gray granite in which the molybdenite, associated with pyrite, occurs as small flakes up to an eighth of an inch in diameter. The writer would judge that the average molybdenite content of the granite was 0.5 per cent, but by careful hand picking ore containing 2 or 3 per cent of molybdenite might be obtained. At the time of the author's visit to the property in the late fall of 1914 the limits of the outcrop of the ore body had not been determined, as in most places the granite was covered with considerable soil, but so far as prospected it appeared to be about 200 by 400 feet. The principal development work consisted of two short prospect tunnels, the relative locations of which are shown in Plate XVII, *B*. The principal constituents of the granite are quartz, feldspar, and biotite. The feldspars have been considerably altered to sericite and kaolin. Accessory minerals are ilmenite, apatite, and zircon. Dr. F. B. Laney, of the United States Geological Survey, who kindly examined samples of ore from this deposit for the author, states that:

> The molybdenite and a small amount of pyrite are distributed irregularly through the rock in such a manner as to suggest the possibility of their being original constit-

uents. In some places the molybdenite occurs in irregular fractures, but is by no means confined to them. Indeed it is distributed pretty well throughout the whole mass of the specimens studied, but appears to be more abundant in the more highly quartzose parts of the rock, thus possibly indicating that it is a late addition to the solidifying or partly solidified magma.

OTHER OCCURRENCES OF MOLYBDENITE IN WASHINGTON.

DEPOSIT NEAR SAFETY HARBOR CREEK, CHELAN COUNTY.

Molybdenite associated with chalcopyrite is reported to occur in Chelan County near the head of Safety Harbor Creek, which flows into Lake Chelan on its northwestern shore about 25 miles in an air line from the foot of the lake. The deposit is situated on the northwestern side of the creek, opposite the Navarre Peaks, and is at an elevation of about 6,000 feet. It is 10 miles over a good trail from the mouth of the creek to the camp. The situation of the deposit is shown in figure 1. The following notes regarding the property were kindly furnished[a] by the owner, P. Robischaud, of Lakeside, Wash. The deposit consists of two systems of veins, one running approximately east, the other north. About 700 feet of development work has been done, mostly in tunnels. The main tunnel is about 330 feet long, and 200 feet of drifts has been run from it. Twelve fissures running east and three running north have been cut. All of these are small, being 18 inches or less in width. The greatest depth reached on any of the veins is about 200 feet. The country rock is said to be porphyry.

Specimens of ore submitted to the bureau consist of a highly altered rock containing molybdenite and chalcopyrite. In most of the specimens the two minerals are not intimately associated, the molybdenite being confined to areas bounding those in which the chalcopyrite occurs; for example, in one piece showing a 4½-inch streak of chalcopyrite the molybdenite occurs in two bands about half an inch wide on either side of the chalcopyrite. The rock in which the sulphides occur has been so changed that determination of its original composition is impossible. The principal constituents are quartz, a carbonate that is seemingly siderite, and a phosphate that is probably apatite.

DEPOSIT ON SHEEP MOUNTAIN, OKANOGAN COUNTY.

J. M. Risley, of Twisp, Wash., has furnished the following information concerning a molybdenite deposit on Sheep Mountain in the northern part of Okanogan County.[b] The property, which consists of five claims, is situated about 1 mile south of Monument No. 11 of the United States-Canadian boundary line and about 20 miles from

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[a] Letter of Nov. 21, 1914.

[b] Letters of Dec. 28, 1914, and July 23, 1915.

Ashnola Siding, a station in British Columbia on the branch line of the Great Northern Railroad to Princeton. The best route to the claim is said to be over a wagon road that runs from this station up the Ashnola River for 8 miles and thence by trail about 12 miles to the property. On the Washington side the deposit is about 55 miles by trail from Twisp.

The deposit consists of three roughly parallel veins 4 to 7 feet wide, the outcrops of which may be traced almost horizontally along the side of the mountain. The veins are well defined and traverse a granite country rock. The altitude of the lowest vein is about 8,000 feet and that of the highest is about 9,000 feet. The two lower veins are below timber line, but the third is above it. Development work consists of several open cuts and short tunnels. Specimens sent to the bureau show the vein material to be a pegmatite in which the molybdenite occurs as flakes up to a quarter of an inch in diameter, associated with molybdite and pyrite. The specimens submitted were free from copper minerals and were estimated to contain about 1 per cent of molybdenite.

PROSPECTS NEAR OROVILLE, OKANOGAN COUNTY.

The author has noted the occurrence of small quantities of molybdenite associated with chalcopyrite in quartz in the Forty-ninth Parallel, Golden Chariot, and O. K. prospects, all of which are situated about 3 or 4 miles north of Oroville, Okanogan County, on the eastern slope of the hills on the west shore of Lake Osoyoos. The mineral also occurs near Concunully in the same county, associated with quartz, sericite, pyrrhotite, and secondary calcite.[a]

MISCELLANEOUS OCCURRENCES.

Other localities in the State in which molybdenite occurs are as follows: Near Skykomish, King County, with chalcopyrite and bornite;[b] in the Monte Cristo district, Snohomish County, as occasional fine flakes;[c] in Pierce County on the north side of Mount Ranier near the White River Glacier at an altitude of about 5,000 feet;[d] on Thunder Creek, a tributary near the head of the Skagett River;[e] in Ferry County on many prospects in the Sanpoil district;[f] also in the Metalline district in Stevens County.[g]

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[a] Specimen in U. S. National Museum.

[b] Specimen in Museum of California Bureau of Mines.

[c] Spurr, J. E., The ore deposits of Monte Cristo, Wash.: U. S. Geol. Survey Twenty-second Ann. Rept., pt. 2, 1901, pp. 777-865.

[d] Letter from Milnor Roberts, University of Washington, Nov. 20, 1913.

[e] Idem.

[f] Sanford, Samuel, and Stone, R. W., Useful minerals of the United States: U. S. Geol. Survey Bull. 585, 1914, p. 205.

[g] Sanford, Samuel, and Stone, R. W., loc. cit.

TABULATION OF MOLYBDENITE AND OF WULFENITE OCCURRENCES IN THE UNITED STATES.

Following are tabulations showing molybdenite and wulfenite occurrences in the United States, with remarks and references to descriptive literature.

Occurrences of molybdenite in the United States.

State.	Remarks.	Reference.
Alaska:		
Jumbo mine, Sulzer	In altered limestone (?) with chalcopyrite.	U. S. Geological Survey Prof. Paper 89, 1915, p. 49.
Treadwell mine	Frequent in ore	Trans. Am. Inst. Min. Eng., vol. 35, 1905, p. 503.
Chilcoot Pass		Specimen in U. S. National Museum.
At mile 192 on Copper River & Northwestern R. R.		Min. and Eng. World, vol. 37, 1912, p. 582.
On Lost River, Seward Peninsula.	Fairly coarse aggregates in iron-stained siliceous gangue.	Specimens in collection of U. S. Geol. Survey.
Arizona:		
See pp. 52 to 58		
California:		
See pp. 58 to 63		
Colorado:		
See pp. 63 to 72		
Connecticut:		
At Haddam, Middlesex County.	In gneiss	Specimen in Brush collection, Yale University; and Dana, E. S., A system of mineralogy, 6th ed., 1911, p. 41.
At Saybrook		Dana, E. S., loc. cit.
South Glastonbury, Hartford County.	In pegmatite	U. S. Geol. Survey Bull. 585, 1914, p. 53.
Idaho:		
At Knob Hill, Kootenai County	On property of the Sauca Consolidated Co.	Min. Ind., vol. 16, p. 723.
Seven Devils district	In Chieftan Copper mine	Specimen in California Bureau of Mines Museum.
At Leadore, Lemhi County	Small flakes with sericite	Specimens in collection of U. S. Geol. Survey.
On south fork of Salmon River, 14 miles from Warren.		Min. Ind., 1905, vol. 14, p. 450.
Maine:		
At Cooper, Washington County.	Flakes in pegmatite dikes and granite.	U. S. Geol. Survey Bull. 340, 1908, pp. 231-240; and U. S. Geol. Survey Bull. 260, 1905, pp. 197-199.
At Brunswick, Cumberland County.	With quartz, feldspar, and biotite.	Specimen in U. S. National Museum; U. S. Geol. Survey Bull. 340, 1908, p. 236.
At Blue Hill Bay and Camdage farm, Hancock County.	In large crystals	Dana, E. S., loc. cit.
At Bowdoinham, Sagadahoc County, and Sanford, York County.		Do.
At Tunk Pond, Hancock County.	Flakes in pegmatite dikes and granite.	U. S. Geol. Survey Bull. 340, 1908, pp. 231-240; Min. World, vol. 31, 1909, pp. 323, 324.
At Buckfield, Oxford County		Eng. and Min. Jour., vol. 82, 1906, p. 1106; U. S. Geol. Survey Bull. 340, 1908, pp. 236-237.
At Greenwood, Oxford County.		Eng. and Min. Jour., vol. 82, 1906, p. 1106.
Mount Mica, 4 miles east of Paris.	In pegmatite	U. S. Geol. Survey Bull. 340, 1908, p. 237.
Massachusetts:		
At Shutesbury, east of Locke's pond.		Dana, E. S., loc. cit.
At Brimfield	With iolite	Do.
At Rockport	In granite	Letter from C. H. Warren, Boston, Mass.
At Quincy	do.	Do.
Minnesota:		
Near Portage, Aitkin County		Min. Res. U. S., 1901, U. S. Geol. Survey, 1902, p. 267.
Montana:		
See pp. 72 to 76		

Occurrences of molybdenite in the United States—Continued.

State.	Remarks.	Reference.
Nevada:		
At Scott's camp, near Alpine		Letter from G. Scott, Alpine, Nev., Nov. 9, 1914.
Lander County		Specimen in Nevada exhibit of Pan.-Pac. International Expo., 1915.
Near Lewis, Lander County		Specimen in California Bureau of Mines Museum.
In Tule Canyon, Esmeralda County.	In quartz	Specimen in Nevada exhibit of Pan.-Pac. International Expo., 1915.
14 miles from Tonopah on Lone Mountain, Esmeralda County.		Eng. and Min. Jour., vol. 76, 1903, p. 667.
At Columbia, Esmeralda County.	With powellite, scheelite, and kaolin.	Specimen in U. S. National Museum.
10 miles west of Winnemucca, on Rose Creek, Humboldt County.		Eng. and Min. Jour., vol. 87, 1909, p. 775.
Winnemucca, Humboldt County.	With quartz, chalcopyrite, and gypsum.	Specimen in U. S. National Museum.
At Mena, Mineral County	Fine grains with molybdenite in siliceous gangue.	Specimens in collection of U. S. Geol. Survey.
26 miles north of Golconda, Humboldt County.	Fairly large aggregates in siliceous gangue.	Do.
Near Amos, Humboldt County.	Fine flakes with quartz sericite, and pyrite.	Do.
Near Yerrington, Lyon County.	Fairly large masses with quartz and feldspar.	Do.
Near head of Death Valley		U. S. Geol. Survey Bull. 585, 1914, p. 120.
In Yerington district, Lyon County.	In pegmatite	Do.
At Redlich, Mineral County	In quartz veins	Do.
At Oak Springs, 45 miles north of Johnnie Siding, Nye County.	With powellite and scheelite	Do.
New Hampshire:		
At Westmoreland	In quartz and feldspar	Specimens in U. S. National Museum and Brush collection, Yale University; Dana, E. S., A system of mineralogy, 6th ed., 1911, p. 41.
At Llandaff	In tubular crystals	Dana, E. S., loc. cit.
At Franconia		Do.
Walpole	With kaolinized feldspar, muscovite, and molybdite.	Specimen in U. S. National Museum.
Warren	With feldspar and hornblende	Do.
New Jersey:		
At Stanhope, Sussex County	In a fine-grained magnetite with massive pyrite in the Hude mine; also in Stanhope mine.	Eng. and Min. Jour., vol. 78, 1904, p. 101; U. S. Geol. Survey Bull. 585, 1914, p. 127.
McAfee	With augite and pyrrhotite in crystalline limestone.	Specimen in U. S. National Museum.
Hackettstown	With chalcopyrite and pyroxene	Specimen in Field Museum of Natural History.
Edison, Sussex County	Crystals in granitoid rock in Ogden mine.	Specimen in American Museum of Natural History.
New Mexico:		
See pp. 77 to 79		
New York:		
In New Bed mines, Essex County.	With pyrrhotite	Trans. Am. Inst. Min. Eng., vol. 27, 1897, p. 199.
In Port Henry iron mines, Essex County.		Do.
Natural Bridge, Jefferson County.		Specimen in California Bureau of Mines.
Lyon Mountain, Clinton County		U. S. Geol. Survey Bull. 585, 1914, p. 138.
West Point, Orange County		Do.
2 miles southeast of Warwick, Orange County.	With rutile, zircon, and pyrite	Dana, E. S., A system of mineralogy, 6th ed., 1892, p. 41.
New York City, near Forty-second Street and First Avenue.	In gneiss	Specimen in American Museum of Natural History.
Tilly Foster mine, Brewster, Putnam County.	With hornblende, augite, chlorite, pyrrhotite, and magnetite.	Specimens in American Museum of Natural History and in Brush collection, Yale University.
Lake Champlain	With quartz, feldspar, and muscovite.	Specimen in U. S. National Museum.
Adirondack Mountains		Min. Ind., vol. 6, 1897, p. 485.

Occurrences of molybdenite in the United States—Continued.

State.	Remarks.	Reference.
North Carolina:		
Demming, McDowell County	With quartz, muscovite-biotite schist.	Specimen in U. S. National Museum.
Near Pioneer Mills mine, Cabarrus County.	In granite and quartz	Genth, F. A., Minerals of North Carolina, 1891, p. 23.
Near Concord, Cabarrus County.	With pyrite in quartz	Dana, E. S., loc. cit.
Guilford County		Hintze, Carl, Handbuch der Mineralogie, vol. 1, 1898, p. 417.
Allegheny County	In Peach Bottom mine	Do.
At Haskett's, Macon County		Do.
West of the Blue Ridge		Do.
Oklahoma:		
Near Roosevelt, in Wichita Mountains.		Eng. and Min. Jour., vol. 77, 1904, p. 398.
Oregon:		
Copper Creek, Eagle Mountains, Baker County.	In white quartz	Bull. University of Oregon, vol. 1, No. 4, 1904, p. 100.
Nine miles from Huntington, down Snake River, Baker County.		Letter from J. L. McKeever, Chicago, Ill., Mar. 28, 1914.
Glacier Park, Middle Eagle Creek.		Bull. University of Oregon, vol. 1, No. 4, 1904, p. 100.
At Drum Lummon mines, in T. 6 S., R. 44 E.	With copper minerals in center of vein 8 feet wide.	Do.
Near Galice, Josephine County.		Min. Ind., vol. 16, 1907, p. 723, and vol. 18, 1909, p. 531.
Pennsylvania:		
Frankford, Philadelphia County	In gneiss, with feldspar, quartz, and biotite; also pegmatite; from Hoffman's quarry, with granular quartz and diopside.	Specimens in Brush collection, Yale University, American Museum of Natural History, and U. S. National Museum; Trans. Am. Inst. Min. Eng., vol. 31, 1901, p. 443.
Germantown	From stone quarries	Proc. of the Academy of Natural Science, 1887, p. 38.
Chester, near Reading	With feldspar, quartz, and muscovite.	Specimen in U. S. National Museum.
Morton, Delaware County	In Johnson's quarry, with feldspar, quartz, and biotite.	Do.
Avondale, Chester County	Leiper's quarry, with feldspar, quartz, and muscovite.	Do.
Rhode Island:		
Copper Mine Hill, Cumberland.	In serpentine matrix with malachite.	Specimen in American Museum of Natural History.
Near Sneech Pond, Cumberland.	In manganese ore	U. S. Geol. Survey Bull. 585, 1914, p. 165.
South Carolina:		
In Haile gold mine, northeast of Kershaw, on Lynches Creek, Lancaster County.	With pyrite carrying gold	U. S. Geol. Survey Bull. 293, 1906, p. 81.
Texas:		
South of Bluffton, Llano County.	In granite, with cyrtolite and fergusonite, with powellite.	Am. Jour. Sci., ser. 4, vol. 38, 1914, p. 485; specimen in U. S. National Museum.
Utah:		
Alta, Little Cottonwood district.	With quartz	Specimen in U. S. National Museum.
In Peruvian Gulch, 1½ miles west of Alta, Salt Lake County.	In siliceous gangue, with pyrite	Specimens in collection of U. S. Geol. Survey.
Near Leamington, Millard County.	Flakes in quartz	Mineral Resources, U. S., 1901, U. S. Geol. Survey, 1902, p. 266.
Vermont:		
At Newport	With apatite	Dana, E. S., loc. cit.
Lexington	With feldspar and quartz	Specimen in U. S. National Museum.
Not given	With kaolinized feldspar and molybdite.	Do.
Washington:		
See pp. 79 to 85		
Wyoming:		
Albany County	With quartz	Field Museum of Natural History.
Encampment, Carbon County		Specimen in California Bureau of Mines Museum.
The Illinois claim of Shoshone Mountain Mining Co., Kirwin.	Flakes in quartz	Letter from Henry Schnitzel, Deadwood, S. Dak., Apr. 3, 1914.

Occurrences of wulfenite in the United States.

State.	Remarks.	Reference.
Arizona:		
See pp. 45 to 52		
California:		
Potrero, San Diego County	In quartz, with malachite and chalcopyrite.	Specimen in California Bureau of Mines Museum.
Lavis, San Bernardino County		Specimen in Museum of Yale University.
Inyo County	In Empire mine	Dana, E. S., A system of mineralogy, 6th ed., 1911, p. 991.
Mount St. Helena, Napa County.	In quartz	Specimen in California Bureau of Mines Museum.
Colorado:		
At Leadville	In ferruginous limestone	Specimen in Field Museum of Natural History.
Massachusetts:		
Southampton	With quartz and sometimes with pyromorphite.	Dana, E. S., loc. cit; specimens in Field Museum of Natural History, American Museum of Natural History, and Brush collection, Yale University.
At Loudville, in Manhan lead mines, Hampshire County.	Sometimes hemimorphic crystals.	Dana, E. S., op. cit., p. 73, and U. S. Geol. Survey Bull. 585, 1914, p. 97.
Montana:		
See pp. 76 to 77		
New Mexico:		
See pp. 77 to 78		
Nevada:		
Eureka District, Eureka County, in Eureka Consolidated and K. K. mines; on Ruby Hill.	With quartz, calcite, and limonite. Possibly of commercial importance.	Trans. Am. Inst. Min. Eng., vol. 6, 1877–78, p. 559; U. S. Geol. Survey Bull. 585, 1914, p. 123; specimens in Brush Collection, Yale University, U. S. National Museum, Colorado State Museum, American Museum of Natural History, and in Field Museum of Natural History.
Tecoma, Elko County	White calcite on limonite	Specimen in American Museum of Natural History and Museum in California Bureau of Mines.
On Comstock Lode, Storey County.		Dana, E. S., op. cit., p. 990.
Redemption mine, Esmeralda County, 1½ miles south of Hornsilver.		U. S. Geol. Survey Bull. 585, 1914, p. 123.
4 miles east of Crescent, Clark County.	With vanadinite	Do.
Quartette mine, Searchlight, Clark County.	With crysocolla and cerussite	Specimen in Harvard collection, Harvard University.
Eldorado mine, Belmont, Nye County.		Specimen in Brush collection, Yale University.
New York:		
Near Sing Sing	With vanadinite and pyromorphite on crystalline limestone.	Dana, E. S., op. cit., p. 991.
Pennsylvania:		
Phoenixville, Chester County	With granite, pyromorphite, anglesite, cerussite, quartz, and galena.	Dana, E. S., loc. cit.; specimens in Brush collection, Yale University; American Museum of Natural History, U. S. National Museum, and Field Museum of Natural History.
Perkiomen, Montgomery County.	With pyromorphite in quartz	U. S. Geol. Survey Bull. 585, 1914, p. 164; specimens in Brush collection, Yale University, and in American Museum of Natural History.
Eaton mines, near Audubon, Montgomery County.		U. S. Geol. Survey Bull. 585, 1914, p. 164.
Pequea mines, Lancaster County.		Do.
South Dakota:		
Richmond mine, Black Hills	With quartz, cerussite, and limonite.	Specimen in U. S. National Museum.
Utah:		
At Alta in the Continental-Alta and City Rocks mines, Little Cottonwood district, Salt Lake County.	In limestone with lead ore	U. S. Geol. Survey Bull. 340, 1907, pp. 239–240.
Lucien district, Box Elder County.	With limestone	Specimen in Field Museum of Natural History and Brush collection, Yale University.

Occurrences of wulfenite in the United States—Continued.

State.	Remarks.	Reference.
Utah—Continued.		
In Tecomah mine		Dana, E. S., loc. cit.; specimen in Brush collection, Yale University.
In Harrington Hickory mine, Beaver County.		Specimen in collection of U. S. Geol. Survey.

PART III. CONCENTRATION.

IMPORTANCE OF SUCCESSFUL CONCENTRATION OF LOW-GRADE ORES.

Successful concentration of the low-grade ores of molybdenum is the one factor on which the future of molybdenum mining depends more than on anything else. Outside of a small and irregular production of wulfenite concentrates, the molybdenum markets of the world have to date been supplied largely by a molybdenite product containing 50 to 90 per cent MoS_2 obtained by the primitive process of cobbing and hand picking high-grade ore. In a few instances hand picking has been supplemented by coarse crushing, preferably in rolls or some similar type of machine that tends to flatten the crystals or flakes of molybdenite, followed by screening to separate the coarser particles of molybdenite from the gangue. That such a method is extremely wasteful is self-evident, and probably in every instance where it has been employed more molybdenite has been thrown on the dump as waste than has been saved. But aside from the inefficiency of the process, the known supply of rich ore on which it can be applied with success, even at the present high price of the molybdenite product, is so small as to promise a yield of only a few tons. Barring the discovery of extensive high-grade deposits of molybdenite, the product obtained by cobbing, hand picking, and screening out the coarser flakes of the mineral will not yield a hundredth part of the tonnage of concentrates requisite to the proper development of the market. Neither will the few known commercial deposits of wulfenite, which is easily concentrated, be able to supply the demand. Low-grade molybdenite ores, of which there are large deposits containing 1 to 3 per cent MoS_2, must therefore be treated.

As yet it has not been proven that any of the chemical or metallurgical processes so far used or proposed for extracting molybdenum are nearly as economical as mechanical processes of concentration when directly applied to low-grade molybdenite ores, particularly if the deposits are situated at a distance from railroad transportation. It appears, therefore, that the mechanical concentration of such ores, including a study of methods for the removal of deleterious elements, such as copper, from the concentrates is the chief problem to be considered. The Bureau of Mines hopes by investigation of these questions to obtain results that will point the way to the establishment of a successful industry.

In this report the question is necessarily taken up in a preliminary way as the bureau has at present no equipment of its own for conducting concentration tests. However, a discussion of the more successful existing methods of concentration is attempted here, and is supplemented by the results of a number of concentration tests of ores selected as representative of typical deposits. The tests described were made possible through the kindness of various companies and individuals who placed the equipment of their testing laboratories at the author's disposal. These tests were of necessity made on laboratory-size machines and on small lots of ore, and the results must be considered only as indicative of what may be expected from the treatment of commercial quantities of ore on machines of standard size.

With proper equipment at its command the bureau hopes to develop improved processes of concentration of these low-grade ores, the results of its work to appear in a succeeding report.

Of the two commercial ores of molybdenum, namely, molybdenite and wulfenite ores, the first are not amenable to the ordinary processes of concentration by jigs, tables, vanners, slimers, etc., such as are usually employed in treating ores of copper, lead, zinc, gold, and silver, as the grains or flakes of molybdenite even when of considerable size float readily on water and therefore are lost if these methods are employed. On the other hand, little difficulty is experienced in the adaptation of the ordinary jig and table processes to wulfenite ores, as they are readily wetted, and because of the high specific gravity (6.7 to 7) of wulfenite it is much heavier than any of the gangue-forming constituents of most of its ores. On account of the wide difference in the character of molybdenite and of wulfenite ores and the necessity of subjecting them to entirely different processes of treatment, the concentration of each is considered separately.

CONCENTRATION OF MOLYBDENITE ORES.

Until recently, practically all the processes of concentration of molybdenite that have been attempted on a commercial scale in the United States have been unqualified failures. A number of companies have erected mills and installed various rolling and screening processes, pneumatic separators, and wet concentrators similar to or adaptations of those usually employed in the treatment of lead, zinc, and copper ores. These mills have operated only a few weeks at the most, and, as far as the author knows, only one company produced more than a ton of marketable concentrates. In several instances, the processes were absolutely unsuited to the ores to which they were applied, and their unsuitability could have been conclusively shown by a trial on an experimental scale. In other instances processes that seemed to promise success from small-scale tests were failures on a commercial scale owing to difficulties in their practical application.

Notwithstanding this record of continuous failures, and although the concentration of molybdenite ore has been considered one of the most difficult problems in ore dressing, there are methods of treatment that have been developed for some time that can be applied to many molybdenite ores with certainty of success. Before proceeding to describe these processes it may be well to state that there is no method suitable for the treatment of molybdenite ores in general, but that each particular ore usually involves its own individual concentration problems; a method or combination of methods that might give highly satisfactory results with one ore might prove an unqualified failure with another. In describing each process the author will attempt to show to which particular type of ores it is adapted, and how some ores are best treated by a combination of processes.

Methods of concentration that have been used with success with molybdenite ores may be grouped under three general heads, as follows: (1) Rolling and screening processes; (2) electrostatic methods; (3) flotation processes. Each of these is considered separately.

ROLLING AND SCREENING PROCESSES.

A considerable proportion of the molybdenite in some ores in which the mineral occurs as large flakes or crystal aggregates may be recovered by crushing the ore in rolls and then screening the crushed material. The success of the process depends entirely upon flattening the molybdenite masses into flakes the maximum diameters of which are in excess of those of the particles of gangue material. Usually before the ore is screened it is passed through a series of rolls to break the gangue into small particles. The material passing through the first screen is again crushed in rolls and screened, and the process repeated as many times as may appear to be advantageous.

About 10 years ago the American Molybdenum Co., of Boston, erected a mill at Cooper, Me., for the recovery of molybdenite by the process outlined above. Hess [a] comments on this plant as follows:

> The plant consisted of a 35-horsepower boiler and engine, a Sturtevant jaw crusher and roll, and four sets of special rolls, each 3 feet in diameter and 10 inches wide. The crusher was but a couple of feet above the floor, from which the material, crushed to about one-fourth inch square, was elevated to the Sturtevant roll, 18 inches in diameter by 4 inches wide, which reduced the ore to about one-eighth inch. It was then elevated to a bin at the top of the building, from which it fell to a series of two special rolls, thence was elevated to a third special roll, and run through a 34-mesh screen. The molybdenite caught on the screen was delivered to a box at the end. The material going through the screen was carried by an elevator and screw conveyor to a fourth roll, from which it fell onto a 40-mesh screen and from that to a 60-mesh screen. What

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[a] Hess, F. L., Some molybdenum deposits of Maine, Utah, and California: U. S. Geol. Survey Bull. 340, 1907, p. 233.

went through the 60-mesh screen was elevated and sent to the tailings pile. It is readily seen that the repeated elevations of the material meant a considerable waste of power. The mill ran only six weeks, and is said to have made about a ton of concentrates, a portion of which seen by the writer was very clean. In the tailings some fine flakes of molybdenite were found, but the amount seemed small. Such a process, if mechanically perfected, might work profitably on deposits where, as in this one, the molybdenite flakes are comparatively broad, but would be wholly unsuited to deposits like many of those in Colorado and elsewhere, in which the individual flakes are of almost microscopic size.

Modifications of the rolling and screening process have been employed at other molybdenite mines both in this country and in Australia. All of them have proved inefficient, and in most instances where they have been used as the sole method of recovery more of the mineral has been thrown on the waste dump than has been saved. In the author's opinion, rolling and screening are of value only as preliminary treatment to remove the coarser and heavier masses of molybdenite from ores in which the mineral occurs in fairly large aggregates. The application of such treatment to this type of ore may be desirable in a few instances, as it saves a considerable part of the molybdenite in coarse form, thereby doing away with losses that would inevitably occur if the large pieces were broken up. How far rolling and screening should be continued depends, of course, on the nature of the ore and on what process of treatment is to follow. In general, however, screening to recover a concentrate should not proceed beyond the point where the succeeding process will begin to handle the material efficiently. Ores containing mica are of course unsuited to this method.

ELECTROSTATIC PROCESSES.

PRINCIPLES OF SEPARATION.

Molybdenite, in common with many other minerals, such as chalcopyrite, pyrite, pyrrhotite, and galena, is a good conductor of electricity as compared with quartz, feldspar, and most other silicates, calcite, etc., which are poor conductors. Hence it may be separated from a majority of the gangue minerals with which it occurs by electrostatic processes. The principle on which these processes are based is that two bodies charged with electricity of similar sign repel each other, but when charged with electricity of opposite signs they attract each other. When minerals that are good conductors of electricity come in contact with a charged body or an electrical field, they almost instantly attain an electrical equilibrium with that body or field and are repelled, whereas as regards poor conductors the time necessary for equilibrium to be reached is so appreciable that if the contact is of short duration the repelling force is negligible. Further, if the mixture of two minerals that are good and poor conductors—for example, molybdenite and quartz—is charged to a high potential with electricity of one sign and placed in contact with a body charged with electricity of opposite sign, the mineral that is a good conductor (molybdenite) almost instantly assumes the charge of the body with which it is in contact, and is repelled. On the other hand, the mineral that is a poor conductor (quartz) is only slowly affected and retains its own charge of opposite sign for an appreciable length of time, and in consequence is attracted to the body with which it is in contact.

RANGE OF APPLICATION.

Theoretically a separation can be effected between any two minerals that differ in degree of conductivity, regardless of whether they are good or poor conductors, but in commercial work it has been impossible to make a separation of good conductors, as there is practically no difference in the time required for them to reach an electrical equilibrium. Therefore, although molybdenite may be separated from quartz, limestone, porphyry, granite, etc., it can not be separated electrostatically from chalcopyrite, pyrite, pyrrhotite, etc. The table following lists the more common minerals and rocks with which molybdenite occurs and shows whether they are good or poor conductors of electricity. In other words, the table indicates whether the mineral mentioned can or can not be separated from molybdenite by electrostatic means. However, it does this in only a general way, as many minerals of varying composition, such as garnet and sphalerite, are sometimes conductors and sometimes nonconductors. Accordingly it is unsafe to predict just what separations may be effected, and the suitability of electrostatic processes for the treatment of any molybdenite ore can be positively ascertained only by actual tests.

Minerals and rocks associated with molybdenite, showing whether they are good or poor conductors of electricity.

Good conductors.	Poor conductors.
Chalcopyrite.	Quartz.
Bornite.	Feldspar.
Chalcocite.	Granite.
Most other copper minerals.	Quartzite.
Pyrite.	Porphyry.
Pyrrhotite.	Garnet.[a]
Magnetite.	Most silicates.
Most other iron minerals.	Calcite.
Galena.	Limestone.
Hornblende.	Sphalerite.[a]
Garnet.[a]	
Sphalerite.[a]	

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[a] Some varieties are good conductors; others are poor conductors.

For the successful application of electrostatic methods to the concentration of molybdenite the ore must be one in which the individual particles of molybdenite are of fair size and in which the gangue and other principal associated minerals are nonconductors. Ores in which the molybdenite is extremely fine to almost amorphous requires fine crushing to free the individual grains from the gangue. Such crushing is not desirable, as it makes dust that usually interferes with the separation and also lowers the capacity of the separators. Thus, the best results are obtained from the treatment of coarse material, so that the ores that are best suited for electrostatic separation are those in which the particles of molybdenite are flaky and of good size, and in which the gangue is clean quartz, granite, etc.

The main requisite in the treatment of the ore is that it be perfectly dry and warm. The necessity of removing all moisture can not be emphasized too strongly, as its presence, even in small quantities, in quartz, feldspar, etc., make these minerals conductors. Ore as coarse as 6-mesh and as fine as 200-mesh may be treated successfully, but, as already stated, the best results are usually obtained with coarse material. Close sizing of the ore is generally unnecessary, but it is apt to improve results, especially in the treatment of coarse material. Dust in the ore should be removed by a blower or other device before the ore is treated.

DESCRIPTION OF SEPARATORS.

The electrostatic separators used in this country are of two main types. One of them consists essentially of a series of metal rods or electrodes charged to a high potential and placed one above the other in an insulated frame. On the top of this frame is a hopper from which the ore is fed by a revolving roll. The ore falls from the roll against the first electrode, and good conductors that come in contact with it are repelled. The residue is thrown against the second electrode by means of a deflector, and more mineral particles that are good conductors are thrown out. The operation is repeated any number of times desired simply by increasing the number of charged rods or electrodes. Generally a standard separating unit of this type consists of a "rougher" and two "cleaners," each having six electrodes. The cleaners are placed back to back and the rougher is placed above them in such a manner that the concentrates from the rougher fall against the first electrode of one cleaner, and the tailings against the first electrode of the other cleaner. In this way the rough concentrates and tailings are re-treated separately. With the exception of the feed roll there is no moving part in the unit, and, consequently, its operation is extremely simple, and the cost of upkeep negligible. High-voltage electricity for charging the electrodes is supplied by stepping up current of ordinary voltage by means of a rotary transformer. Thus the operation of the separator is independent of weather conditions, which so noticeably affect the operation of electrostatic generators.

The other type of separator has a revolving metal electrode onto which ore is fed. Opposite and adjacent to it is another electrode consisting of a row of sharp metal points. The first electrode is grounded, and the second is connected to one pole of an electrostatic generator, the other pole of which is grounded. The electricity delivered to the pointed electrode is discharged by convection to the grounded electrode and is dissipated as fast as received. The revolution of the grounded electrode carries the mineral particles through the convective discharge and results in their receiving a charge. Good conductors immediately lose this charge to the grounded electrode on which they rest; consequently they are neither attracted nor repelled by the electrode but simply fall off of it as it revolves. On the other hand, mineral particles that are poor conductors accumulate a charge on their surfaces nearest the pointed electrode, and on account of their nonconducting character they are polarized and are caused to adhere to the revolving electrode. These particles are brushed off the back of the electrode by a felt roller.

Three of the separating units described above are generally combined in one machine for commercial work in such a manner as to provide for three successive treatments of the mineral particles that are good conductors. In the separation of molybdenite and quartz, for example, a rough concentrate would be made on the first unit and it would be further cleaned on the second and third units.

This type of separator is open to the objection that it is dependent on electrostatic generators for its supply of electricity, and in moist climates or wet weather these give much trouble in their operation.

CONCENTRATION TESTS.

The tests described below were made on molybdenite ore submitted by the Santa Maria Molybdenum Mining & Milling Co., of San Diego, Cal. The ore, which was said to be a representative sample from this company's property 40 miles east of that city, was an iron-stained granite containing about 0.5 per cent of molybdenite and a small quantity of pyrite. The molybdenite occurred in radiating masses from the size of a pea to an inch or more in diameter. Microscopic examination showed that the granite was composed of quartz, orthoclase, plagioclase varying from albite to oligoclase, biotite, and a few minor accessory minerals. This ore was selected for these tests as in a general way it was typical of ores from a large number of deposits.

On account of the low grade of the ore it would be necessary to hand pick it in a commercial operation, and therefore, previous to crushing for electrostatic separation, a hand-picking test was made of the sample submitted. The results of this test are given in the following table:

Results of hand picking molybdenite ore from the Santa Maria Molybdenum Mining & Milling Co.'s property, 40 miles east of San Diego, Cal.

Item.	Original ore.	Sorted ore.	Reject.
Weight, pounds	313.62	115.62	198.00
Per cent MoS_2	[a].47+	1.15	.08
Weight MoS_2, pounds	1.488	1.330	.158
Extraction MoS_2, per cent		89.38	
Loss MoS_2, per cent			10.62

[a] Calculated.

The lot of ore, which weighed approximately 313 pounds, was cobbed down to pieces 2 inches in maximum diameter, and hand picked. One hundred and ninety-eight pounds of the material was discarded and the remaining 115 pounds of selected ore was crushed in a laboratory-size jaw crusher to pass a 10-mesh screen. The discarded ore was also crushed through a 10-mesh screen, and both products were sampled and assayed. They contained 1.15 and 0.08 per cent MoS_2. The ratio of concentration obtained was approximately 1 to 3, and the loss of molybdenite in the discarded material about 10 per cent.

For the electrostatic tests the hand-picked ore, which had been crushed through a 10-mesh screen, was screened into three sizes—through 10-mesh on 20-mesh, through 20-mesh on 60-mesh, and through 60-mesh. The coarser size was treated on a Sutton, Steele & Steele type of concentrator and the two finer sizes on a concentrator of the Huff type. The results of these tests are given in the following table:

Results of electrostatic-concentration tests of hand-picked molybdenite ore from the Santa Maria Molybdenum Mining & Milling Co.'s property, 40 miles east of San Diego, Cal.

THROUGH 10-MESH ON 20-MESH SUTTON, STEELE & STEELE TYPE CONCENTRATOR.

Item.	Ore.	Concentrates.	Middlings.	Tailings.
Weight, pounds	30.35	0.48		29.87
Per cent MoS_2	[a]1.58	87.17		.21
Weight MoS_2, pounds	.481	.418		.063
Extraction MoS_2, per cent		86.90		
Loss MoS_2, per cent				13.10

[a] Calculated.

Results of electrostatic-concentration tests of hand-picked molybdenite ore from the Santa Maria Molybdenum Mining & Milling Co.'s property, 40 miles east of San Diego, Cal.—Continued.

THROUGH 20-MESH ON 60-MESH HUFF TYPE CONCENTRATOR.

Item.	Ore.	Concentrates.	Middlings.	Tailings.
Weight, pounds	41.49	0.52	0.10	40.87
Per cent MoS_2	[a]1.26+	76.66	41.87	.20
Weight MoS_2, pounds	.523	.399	.042	.082
Extraction MoS_2, per cent		76.29	8.03	
Loss MoS_2, per cent				15.68

THROUGH 60-MESH HUFF TYPE CONCENTRATOR.

Weight, pounds	37.18	0.36	0.45	36.37
Per cent MoS_2	[a].94+	46.78	23.63	.21
Weight MoS_2, pounds	.350	.168	.106	.076
Extraction MoS_2, per cent		48.00	30.29	
Loss MoS_2, per cent				21.71

SUMMARY.

Total weight, pounds	109.02	1.36	0.55	107.11
Total weight MoS_2, pounds	1.354	.985	.148	.221
Per cent MoS_2	[a]1.24+	[a]72.42+	[a]26.90+	[a].20+
Extraction MoS_2, per cent		72.75	10.93	
Loss MoS_2, per cent				16.32

[a] Calculated.

It will be noted that with the coarser material a concentrate containing 87.17 per cent MoS_2 was made, and that the recovery in these concentrates was 86.9 per cent. The concentrates from the next size contained 76.66 per cent MoS_2, and accounted for 76.29 per cent of the molybdenite in the feed. However, a middling product containing 41.87 per cent MoS_2 was made, which increased the total extraction on this size of material to 84.32 per cent. The fine ore yielded concentrates and middlings that assayed 46.78 per cent and 23.63 per cent MoS_2, and accounted for an extraction of 78.29 per cent. It will be noted that the best work was done on the coarse material, both as regards recovery and grade of concentrates.

A summation of the three tests shows that the average grade of the products made was as follows: Concentrates, 72.42 per cent; middlings, 26.9 per cent; and tailings, 0.20 per cent MoS_2. The total recovery on the concentrates and middlings was 83.68 per cent. The results obtained were satisfactory when it is considered that the ore treated contained pyrite, which of necessity was concentrated with the molybdenite.

SUGGESTED METHOD FOR TREATMENT OF A TYPICAL ORE.

For ores of the same general type as that mentioned above—that is, ores in which the individual particles of molybdenite are flaky and of fair size and the gangue is clean quartz, granite, or other nonconductor—the following general outline of the proper method of treatment is suggested:

The original ore, or selected ore, if hand picking is advisable, should be crushed in a jaw or gyratory crusher to about one-half inch to 1-inch size and the product crushed in rolls to pass a 6-mesh screen. This material should then be treated in a direct-heat rotary drier until all moisture has been removed, and screened to sizes approximately as follows: Through 6-mesh on 12-mesh; through 12-mesh on 20-mesh; through 20-mesh on 40 or 60 mesh; through 40 or 60 mesh. If much dust is present in the finest size of material, its removal with some sort of blower or other device previous to electrostatic treatment will probably be advantageous. Each size of the dried material should be treated separately on whatever type of electrostatic concentrator has been selected. The best method of operating these separators depends, of course, on their type, and also on the character of the ore being treated. In the author's opinion it is generally advantageous to operate the concentrators so as to obtain as clean tailings as possible. In this way the bulk of the material is eliminated at once, and the re-treatment of the concentrates and of the middling product takes much less time than that required in re-treating the original tailings. This method of operation will generally yield a better grade of concentrates and a higher recovery than any other. Also, the grading of the concentrates and middlings so as to obtain the best prices for the combined products can be more readily accomplished by this method. In some instances the middling products from the treatment of the coarser sizes of ore consist largely of particles of gangue with included grains of molybdenite. Under such conditions, the middlings should be crushed, sized, and re-treated.

To the author's knowledge, there are no plants that are treating molybdenite ore commercially by electrostatic methods; hence, figures as to costs of operation are unavailable. It seems reasonable to infer, however, that they would be only slightly different from those for treating zinc or other ores to which electrostatic methods have been successfully applied. The costs of drying and separating zinc-iron concentrates by electrostatic methods, including only operating costs, do not usually exceed $1 per ton of material treated; and the total costs, including operation, royalty, taxes, and all other overhead expenses, such as interest on investment and amortization charges, usually range from $2.50 to $3.50 per ton. The costs of treating molybdenite ore would probably be slightly in excess of these figures, as the capacity of the separators would, no doubt, be less than when operating on zinc-iron concentrates, where the weight of concentrates and tailings produced is more nearly equal than it would be with the usual molybdenite ores. Drying of the molybdenite ore would probably cost 10 to 20 cents per ton, depending on the size and type of drier used, the cost of fuel and labor, and the percentage of moisture in the ore.

If the electrostatic separators were so arranged that the products could be taken care of automatically, one man could tend 8 to 10 concentrators. The other labor required for operating a mill capable of treating 50 tons of crude ore per day would probably not exceed four or five men per shift, and no high-priced labor would need to be employed on account of the simplicity of the process.

The author is confident that electrostatic processes will yield good results with all molybdenite ores to which they are suited and to which they are intelligently applied, and they are of particular value for treating ore that can not be treated by flotation methods on account of the absence of requisite water supplies.

FLOTATION PROCESSES.

Molybdenite has the property, common in varying degree to most metallic sulphides, such as chalcopyrite, sphalerite, galena, pyrite, and pyrrhotite, of not being wetted readily by water, and, when dry and in small particles, of floating on a water surface. Moreover, like those sulphides, it is easily wetted by most oils. Further, in a pulp of crushed ore and water, oils have a preferential wetting action for particles of molybdenite as against particles of gangue minerals such as quartz, and this selective wetting action is decidedly increased if the water is slightly acidified. Particles of molybdenite so wetted with oil are covered with a buoyant water-repelling coating that materially assists their flotation. As the reasons for many of these phenomena—for example, the selective wetting action of oils and the increase of this selective action by acids—are not clearly understood, and as even an elementary discussion of the accepted theories of mineral flotation would be out of place here, the reader who wishes further information on the subject is referred to a clear and concise exposition by Hoover[a] and to an excellent paper by Mickle.[b] It suffices here to say that the phenomena mentioned above are the basis on which all flotation processes depend. In many processes the area of the effective surface of flotation is increased by the liberation of bubbles of gas or air in the liquid, the surface of each bubble acting in the same way as the horizontal surface of a liquid at rest. These bubbles may be of air and may be produced by violent agitation of the pulp or by release of air from solution in the liquid by a reduction of pressure, or they may be of carbonic-acid gas formed by the action of sulphuric acid on limestone or other carbonates or by other means.

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[a] Hoover, T. J., Concentrating ores by flotation, 1912, 221 pp.

[b] Mickle, K. A., Flotation of minerals: Proc. Royal Soc. Victoria, vol. 23, pt. 2, 1911, pp. 555-585, abstracted by Eng. and Min. Jour., vol. 92, 1911, pp. 307-310, and vol. 94, 1912, pp. 71-76.

WATER-FLOTATION PROCESSES.

CHARACTER OF WATER FLOTATION.

Water flotation for the concentration of molybdenite depends solely on the fact that small dry particles of the mineral float readily on water, whereas the usual gangue material is easily wetted and sinks. They do not of necessity involve the use of oil, acid, or gas, and their application is extremely simple. The concentrators, which are of various types, consist essentially of a device for feeding the crushed ore in as thin a sheet and at as uniform a rate as possible onto a moving water surface in a tank and an arrangement, either by an overflow or a revolving belt of canvas or other suitable material, for discharging the floating film of concentrates into another tank. The tailings are usually drawn off from a spigot in the bottom of the first tank. In some types of apparatus the ore from the feeder is allowed to slide down an inclined plane or concave, over which a film of water is passed and from which the ore is discharged approximately in the plane of the water surface in the tank. In other apparatus the ore from the feeder falls on top of an almost submerged corrugated or canvas roller, the revolution of which carries the ore forward to the flotation surface. The object of all these devices is to place the ore on the water in a sheet only one mineral particle in depth with as little disturbance of the water surface as possible and with the majority of the particles of gangue already wetted.

Even with the best of feeding devices some particles of gangue fall on the floating film of concentrates or are otherwise mechanically entrained by it. Various methods of cleaning the film, such as allowing it to flow down an incline into a second tank, picking it up on a roller or belt and again discharging it to a flotation surface and dividing it into a large number of parts and agitating it by causing it to flow through the teeth of a comblike obstruction, are used.

No description of any particular water-flotation concentrator is attempted here, as detailed information both as to the design and the operation of a number of different types of machines is given in the references cited below.[a]

NECESSARY FACTORS IN SUCCESSFUL WATER FLOTATION.

To be concentrated successfully by water flotation, a molybdenite ore should be such as to require only medium-fine crushing to liberate the molybdenite, and the gangue should be one in which the individual particles are readily wetted. Further, if a high-grade concentrate is to be obtained the ore must be practically free from other sulphides such as pyrite, pyrrhotite, and chalcopyrite, which would be concentrated with the molybdenite. Proper treatment of the ore previous to flotation is of great importance. It should be reduced to approximately 10-mesh, or as much finer as may be necessary to liberate the mineral from the gangue, by crushing in rolls in such a manner as to make the quantity of fines as small as possible. Then if it is at all damp it should be thoroughly dried. Mechanical difficulties in the proper feeding of fine ore, and the great reduction in the capacity of the concentrators when fine material is treated exclusively, render advantageous in most instances the treating of the ore without sizing, although the capacity of the concentrators and the grade of concentrates made are considerably increased when coarse material that has been sized is treated.

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[a] Wood, H. E., The Wood flotation process: Trans. Am. Inst. Min. Eng., vol. 44, 1912, pp. 684–701; Concentration of molybdenite ores: Eng. and Min. Jour., vol. 93, 1912, pp. 227–228.

The capacity of water-flotation machines varies with their type and with the character of the ore treated, and depends directly on the size to which the ore is crushed, the amount of fines made, the nature of the gangue, and the ratio of concentrates to tailings. In speaking of a particular type of concentrator, Wood [a] says:

> A standard machine treating a 20-mesh quartz ore, using a 3-foot width of feed and having a 4-foot take-off belt, will vary in capacity from 1,000 to 2,000 pounds per hour, unless the ratio of concentration is low, in which case the capacity will be smaller. Some ores that possess an easily wetted gangue and call for a high concentration ratio can be fed rapidly at 20-mesh, 30-mesh, or 40-mesh. For instance, a 1 or 2 per cent molybdenite ore in a quartz gangue will give a clean concentrate, even if the ore is fed several times faster than an ordinary sulphide ore.

The writer thinks that the capacities stated above could be had only at a sacrifice of either the recovery or the grade of concentrates obtained, and that a machine with a feed 3 feet wide, handling 300 to 500 pounds per hour of ore crushed to pass a 20-mesh screen would be treating about the maximum quantity of material that it could separate efficiently. No figures as to cost of operation can be given, but they are presumably small as the concentrators require only moderate quantities of water and little power.

RESULTS OF TESTS WITH WOOD'S PROCESS.

The accompanying table shows the results obtained by Wood in 10 concentration tests of five different molybdenite ores. In every test except the last, in which the ore had been slightly roasted, the recoveries were good, averaging nearly 90 per cent. In general, however, the grade of concentrates was low to medium, averaging less than 60 per cent MoS_2. Test No. 3 is of special interest as it shows the results obtained by flotation of the same ore on water at different temperatures. The decided improvement in the grade of concentrates by using warm water may be attributed to the decreased surface tension of the liquid allowing some particles of gangue to sink more readily. The recovery with warm water is, however, remarkable and can be accounted for only by some factor, such as a difference in the rate of feed, that would make the tests not strictly comparable. Detailed information as to the tests is given by Wood.[a] A summary of the data obtained by Wood is given in a table which is presented below:

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[a] Wood, H. E., The Wood flotation process: Trans. Am. Inst. Min. Eng., vol. 44, 1912, p. 693.

Results of concentration tests of molybdenite ores by Wood flotation process.

Test No.	Size of feed.	Per cent MoS_2.		Recovery, per cent.	Remarks.
		In feed.	In concentrates.		
1	Through 40-mesh..	2.08	41.38	99.00	Colorado ore. Retreatment would materially increase the grade of concentrates with little loss.
2	Through 20-mesh..	2.00	61.55	92.27	Alaskan ore.
3	....	1.87	45.31 (*a*) 56.70 (*b*)	79.08 (*a*) 89.17 (*b*)	Canadian ore with a quartz and mica gangue. (*a*) was obtained with water at 56° F. and (*b*) with water at 110° F.
4	Through 20-mesh..	6.66	63.50	96.34	Foreign ore.
	Through 30-mesh..	6.73	67.42	86.26	
	Through 40-mesh..	6.66	65.50	86.48	
	Through 40-mesh..	8.95	74.60	94.19	
5	Through 40-mesh..	8.65	30.00 (*a*) 81.45 (*b*)	79.00 (*a*) 26.30 (*b*)	Alaskan ore containing pyrrhotite and magnetite, and low in silica. (*a*) was obtained with raw ore, and (*b*) with slightly roasted ore.

In order to obtain data as to concentration of a molybdenite ore containing copper sulphides by this process a small-scale test was made of a sample of ore from the Whale claim, in Copper Canyon, near Copperville, Ariz. The ore consisted of a clean white quartz containing about 7 per cent of molybdenite and 2 per cent of copper, present largely as chalcopyrite.[b]

After the ore had been dried and then crushed in a ball mill to pass a 40-mesh screen, it was concentrated on a laboratory-size flotation machine of the Wood type. The concentrator was so arranged that the floating film of concentrates picked up by the take-off belt was discharged to a second flotation surface from which it was allowed to run into a concentrate tank. The material that sank in the second flotation tank was considered middlings. The ore and water feeds to the machine were adjusted with the idea of causing as much chalcopyrite as possible to sink. The results of the test are given in the accompanying table.

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[a] Wood, H. E., The Wood flotation process: Trans. Am. Inst. Min. Eng., vol. 44, 1912, pp. 684–701.

[b] For a detailed description of this ore see p. 52.

Results of water-flotation test with concentrator of the Wood type of molybdenite-chalcopyrite ore from the Whale claim, near Copperville, Ariz.

Item.	Ore.	Concentrates.	Middlings.	Tailings.
Weight, grams	4,000.00	538.00	107.00	[a] 3,355.00
Per cent MoS_2	[b] 7.10+	47.44	6.74	.65
Per cent Cu	[b] 1.93+	5.26	5.90	1.27
Weight MoS_2, grams	284.25	255.23	7.21	21.81
Weight Cu, grams	77.22	28.30	6.31	42.61
Extraction MoS_2, per cent		89.79	2.54	
Extraction Cu, per cent		36.65	8.17	
Loss MoS_2, per cent				7.67
Loss Cu, per cent				55.18

[a] 467 grams of material lost in the overflow was assumed to assay the same as the 2,888 grams of tailings recovered.

[b] Calculated.

The great difference between the percentage of recovery of molybdenite and of chalcopyrite is of particular interest. As compared with the feed, the concentrates contained approximately seven times the percentage of MoS_2 and less than three times the percentage of copper, and the recovery in the concentrates was 89.79 per cent of the molybdenite, as compared with only 36.65 per cent of the copper. Inasmuch as the crushed ore had stood only a short time before treatment, so that the particles of chalcopyrite had little opportunity to oxidize, which would cause them to be more readily wetted, the author considers the widely different results obtained in the concentration of the two minerals to be remarkable.

It is of interest to note that a water-flotation process for the concentration of molybdenite ore is being used at present on a commercial scale by Henry E. Wood & Co., of Denver, Colo.

Methods of treating both the original ore and the concentrates to remove pyrite, chalcopyrite, pyrrhotite, and other metallic sulphides and such ingredients as mica that are likely to float with the molybdenite are discussed later.

OIL-FLOTATION PROCESSES.

Many of the principal processes of concentration by oil flotation are described in detail by Hoover[a] and in numerous articles that have recently appeared in the mining press. Published data giving the results obtained by the application of these processes to molybdenite ores are extremely meager, and are practically confined to the results of treatment by the Elmore vacuum flotation system. As the Bureau of Mines at present has no equipment at its disposal for conducting tests of the various flotation processes, the concentration of molybdenite by these methods must, of necessity, be discussed here in only a general way.

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[a] Hoover, T. J., Concentrating ores by flotation, 1912, 221 pp.

Broadly speaking, oil flotation probably offers the best method of treating molybdenite ores in general, and those particular processes that have been successfully applied in concentrating ores of the other metallic sulphides would, without doubt, meet with equal success in the treatment of molybdenite. The above statement does not mean that all molybdenite ores can be successfully concentrated by oil flotation, for the physical characteristics and mineralogical composition of an ore are as important factors in determining the success of any oil-flotation process as of electrostatic or water-flotation methods. The ores best suited for treatment by oil flotation are those in which the molybdenite is flaky. From ores in which the mineral is so fine as to be almost amorphous and the gangue contains a considerable proportion of soft material, such as kaolinized feldspar, it is almost impossible to obtain a good grade of concentrates because of the flotation of some of the finer particles of gangue. Of course, if molybdenite is associated with other metallic sulphides, such as chalcopyrite, pyrite, or pyrrhotite, these are concentrated too, and must either be removed from the ore, by a preliminary treatment, or from the concentrate if a high-grade product is desired. Methods that may be employed for this purpose are discussed later. Ores containing magnetite, hematite, garnet, hornblende, or similar gangue minerals that are good conductors of electricity, and therefore are not adapted for concentration by electrostatic methods, are especially suited for treatment by flotation.

Laboratory experiments indicate that in general the particles of molybdenite that can be floated are much coarser than those of other metallic sulphides, and if finer crushing is not necessary for the liberation of the molybdenite from the gangue, material as coarse as 20 mesh may be successfully concentrated. On the other hand, there is every reason to believe that a good recovery and a fair grade of concentrates can be obtained from the treatment of molybdenite through 200 mesh because galena and sphalerite of similar fineness have been successfully treated by oil flotation. In general, therefore, oil-flotation processes are applicable to a wider range of sizes than are electrostatic or water-flotation methods, and many of them have the added advantage of large capacities from small units. For example, Hoover estimates that in one type of apparatus used in the minerals-separation process a unit consisting of six mixing boxes each only 16 inches wide and 36 inches deep will have a daily capacity of 50 to 60 tons of ore,[a] presumably Broken Hill lead-zinc tailings, and a 5-foot Elmore machine will ordinarily treat from 25 to 45 tons of crude ore in 24 hours.[b] Capacities equal to and perhaps even greater than these could probably be obtained in the treatment of molybdenite ores by the same units, but no definite data either as to capacities or as to costs of any oil-flotation process when applied to molybdenite ore have, to the author's knowledge, been made public. In fact, owing to the policy of secrecy adopted by most companies interested in the development of oil-flotation methods, little information is available regarding the economics of the various processes, even as more commonly applied to the concentration of the sulphides of copper, lead, and zinc. However, comprehensive data with regard to the costs of treating Broken Hill lead-zinc tailings by four well-known processes, namely, the Potter-Delprat, De Bavay, Elmore, and minerals separation, are given by Hoover.[a] His estimates of 3s. 6d. ($0.85) per ton as the average cost of flotation and 9s. 3d. ($2.25) per ton as the average total cost of re-treating these tailings on a large scale form a poor basis on which to estimate the cost of treating molybdenite ores by the same processes in small plants such as might be installed at mines producing 50 or 100 tons of ore a day. It is safe to say, however, that the costs of small-scale concentration of molybdenite ores would be considerably higher than those given above.

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[a] Hoover, T. J., Concentrating ores by flotation, 1912, p. 120.

[b] Hoover, T. J., op. cit., p. 101.

RESULTS WITH ELMORE VACUUM FLOTATION.

The following table shows the results obtained in concentration tests of two molybdenite ores by the Elmore vacuum-flotation system. It will be noted that in the first test, in which the gangue was largely feldspar, the percentage recovery was high and the concentrate obtained was only medium grade, probably because of the flotation of some of the finer particles of light feldspar. In the second test, in which the gangue was relatively heavy, chiefly magnetite and garnet, both the grade of concentrates and the recovery obtained were materially better.

Concentration tests on molybdenite ore by the Elmore vacuum-flotation process.[a]

Per cent MoS_2.			Recovery, per cent.	Remarks.
In ore.	In tailings.	In concentrates.		
5.67	0.42	68.00	93.2	Gangue principally feldspar.
3.83	.10	85.95	98.1	Gangue principally magnetite and garnet.

[a] Converted from figures given by A. S. Elmore, Vacuum flotation process for concentration: Eng. and Min. Jour., vol. 83, 1907, pp. 908, 909.

It is reported that the Elmore process is being used for treating molybdenite at two mines in Norway and that large quantities of concentrates have already been shipped.[b] The writer believes that these plants are as yet the only ones in which any type of oil concentration has been attempted for concentrating molybdenite on a commercial basis.

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[a] Hoover, T. J., op. cit., pp. 164-166.

[b] Molybdenum recovery by Elmore process: Eng. and Min. Jour., vol. 99, 1915, p. 907.

The results of several large-scale concentration tests by the Ore Concentration Co. (Ltd.), of London, of four different lots of molybdenite ore by the Elmore process are given in the accompanying table. The results of these tests, which were made with ore crushed through 30 mesh, compare favorably with those given in the preceding table and indicate in general what might be expected in the treatment of any molybdenite ore to which the process is adapted.

Concentration tests of molybdenite ore by the Elmore vacuum-flotation process.[a]

Weight of ore treated, tons.	Per cent MoS_2 in concentrates.	Recovery, per cent.
32.0	85.68	91.5
32.0	86.30	94.5
37.0	84.25	89.7
3.5	83.00	90.0
104.5	84.80	91.7

[a] Molybdenum recovery by Elmore process: Eng. and Min. Jour., vol. 99, 1915, p. 907.

REMOVAL OF ACCOMPANYING METALLIC SULPHIDES.

When other metallic sulphides, such as chalcopyrite, pyrite, or pyrrhotite, accompany molybdenite they are concentrated with the latter mineral by both electrostatic and flotation methods. The presence of pyrite or pyrrhotite in the concentrates is undesirable only in that it lowers the grade of the product, but chalcopyrite, bornite, or other copper sulphides are objectionable in that if the concentrates contain more than 1 or 2 per cent of copper they are practically unsalable, and even small percentages of copper greatly lower their value.

Various methods have been suggested for removing the other sulphides either before or after the concentration of the molybdenite. Most of these methods depend on the ore or concentrates being lightly roasted. In this way the larger part of the pyrite and chalcopyrite are rendered magnetic and may be removed by means of a magnetic separator, or, through the formation of a coating of oxide on the surface of each particle, the pyrite and chalcopyrite in the ore are readily wetted and sink if the material is subjected to flotation. It is at once evident that these new properties imparted to the pyrite and chalcopyrite by roasting can be taken advantage of either separately or in a combination of processes. In the writer's opinion it is cheaper and more effective as regards ultimate recovery of molybdenite to remove the other sulphides after concentration rather than before. No matter how carefully conditions of roasting are controlled some of the molybdenite will be partly converted to oxide on the surface of the individual grains, and this coating will entail some loss of the molybdenite whether the material is treated by flotation or on an electrostatic separator. So far as recovery of the molybdenite is concerned it is, therefore, advisable to roast the combined concentrates and remove the oxidized pyrite and chalcopyrite by magnetic separation.

On the other hand, even with the most carefully regulated roasting, all the pyrite and chalcopyrite do not become magnetic. If the nonmagnetic product from the magnetic separator contains an appreciable amount of copper its retreatment by flotation may be advisable in order to further rid the molybdenite from grains of chalcopyrite and pyrite which, although nonmagnetic, are covered with a film of oxide that will cause them to become wet and to sink. In other cases it may be advisable to dispense entirely with magnetic separation and to treat the lightly roasted concentrate directly by flotation.

A method for separating chalcopyrite, which takes advantage of the oxidation of the surface of the particles at ordinary temperatures, is suggested by a correspondent of the Engineering and Mining Journal. In regard to a molybdenite ore containing chalcopyrite, he states that: "After grinding the ore to 40 mesh, it was dampened and dried. Under these conditions 90 per cent of the MoS_2 will float, and practically every particle of the chalcopyrite sinks. This furnishes a cheaper method than to roast the mixed flotation concentrates with the intention of removing the copper ore magnetically."[a]

Wood suggests treating the combined concentrates on the ordinary type of wet concentrating table with the idea of separating the molybdenite in what is usually the tailing product.[b] The author believes that this method would not be particularly effective. Moreover, inasmuch as the presence of even a small fraction of 1 per cent of copper is objectionable in the concentrates, it is doubtful whether any of the processes suggested above are commercially capable of reducing the copper to 0.1 per cent., for example, in any material in which the ratio of copper sulphide to molybdenite is relatively large. For such ore chemical methods of separation will probably have to be used.

SUMMARY.

Each particular molybdenite ore presents its own concentration problems. Usually one of the methods, or a combination of the methods, discussed above will give satisfactory results, but in some instances other ore-dressing processes must be employed in conjunction with them.

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[a] Molybdenum, tungsten, and uranium ores, Eng. and Min. Jour., vol. 97, 1913, p. 114.

[b] Wood, H. E., The Wood flotation process: Trans. Am. Inst. Min. Eng., vol. 44, 1912, p. 693.

CONCENTRATION OF WULFENITE ORES.

Unlike the concentration of molybdenite, the treatment of wulfenite ores presents few difficulties. Not only are the ordinary wet processes of concentration by jigs, tables, vanners, slimers, etc., such as are usually employed in the treatment of ores of lead, copper, and zinc, adapted to the concentration of wulfenite, but, when properly applied, they result in a high extraction of the mineral. Moreover, even the finest particles of wulfenite are readily wetted; hence sliming of the ore does not occasion the considerable losses that would occur under similar circumstances in the treatment of most metallic sulphides, such as galena and chalcopyrite, as these minerals float readily when finely divided. On account of the high specific gravity of wulfenite (6.7 to 7) it is readily separated from all of the gangue minerals with which it occurs. It is, however, frequently associated with vanadinite (specific gravity, 6.66 to 7.23), from which it can not be separated by wet methods, also with cerussite (specific gravity, 6.46 to 6.57), and anglesite (specific gravity, 6.12 to 6.39), from which it can be only partly separated. Occasionally small quantities of other lead minerals of high specific gravity, such as galena (specific gravity, 7.4 to 7.6), pyromorphite (specific gravity, 6.5 to 7.1), and mimetite (specific gravity, 7.0 to 7.25), occur with wulfenite and are then recovered in the wulfenite concentrate. The presence or absence of these other heavy minerals in the ore determines largely the grade of wulfenite concentrates that can be made. Theoretically, a concentrate consisting entirely of pure wulfenite contains 39.23 per cent molybdic trioxide (MoO_3), and with many ores a product with 34 to 36 per cent molybdic trioxide is easily obtained, whereas with others a concentrate containing even 20 per cent molybdic trioxide is obtained with difficulty owing to the presence of considerable percentages of vanadinite, cerussite, etc. The costs of concentrating wulfenite ores by wet processes are in general comparable to those of treating ores of galena, sphalerite, chalcopyrite, etc., by similar methods.

Many deposits of wulfenite ore are situated at considerable distances from water supplies of sufficient magnitude to furnish the quantities requisite for wet concentration. It is, therefore, of interest to note that wulfenite can be concentrated successfully by pneumatic processes, and that there are pneumatic separators of various types, such as tables and jigs, now on the market that without doubt can effect nearly as high an extraction and make as good grade of concentrates as can be obtained by wet methods. The cost of separation by pneumatic processes is, of course, greater than by wet methods.

CONCENTRATION TESTS OF WULFENITE ORE FROM OLD YUMA MINE, NEAR TUCSON, ARIZ.

The tables following give the results obtained by the author from a series of concentration tests of wulfenite ore from the Old Yuma mine, 14 miles northwest of Tucson, Ariz. The tests were not conducted with the idea of deriving the best method of treatment for the ore in question, but to show the wide range of processes that may be successfully applied to wulfenite ores in general. The ore used was of high grade, containing approximately 6 per cent molybdic trioxide. The gangue was an iron-stained silicous rock in which the wulfenite was present in crystals varying in size from minute specks up to one-half inch in maximum diameter. A sample of the original ore weighing 120 pounds was crushed in a small jaw crusher to pass through a 3-mesh screen, and divided into six lots by careful sizing. The first three of these lots, embracing material through 3-mesh on 20-mesh, were treated in a hydraulic pulsator jig. The next two lots, embracing material through 20-mesh on 100-mesh, were concentrated in a pneumatic jig, and the last lot of material through 100-mesh was run on a concentrating table of common make.

Concentration tests, on machines of various types, of wulfenite ore from the Old Yuma mine, 14 miles northwest of Tucson, Ariz.

TEST 1. CONCENTRATION ON HYDRAULIC PULSATOR JIG.

Item.	Through 3-mesh, on 6-mesh.				Through 6-mesh, on 10-mesh.			
	Ore.	Concentrates.	Middlings.	Tailings.	Ore.	Concentrates.	Middlings.	Tailings.
Weight, pounds	32.50	3.54	1.46	27.50	27.00	4.73	1.00	21.27
Per cent MoO_3	[a]4.59+	36.43	8.46	.29	[a]6.14+	32.76	5.13	.37
Weight MoO_3, pounds	1.494	1.290	.124	.080	1.680	1.550	.051	.079
Extraction MoO_3, per cent		86.35	8.30			92.26	3.04	
Loss MoO_3, per cent				5.35				4.70

Item.	Through 10-mesh, on 20-mesh.				Total.			
	Ore.	Concentrates.	Middlings.	Tailings.	Ore.	Concentrates.	Middlings.	Tailings.
Weight, pounds	18.00	4.55	1.57	11.88	77.50	12.82	4.03	60.65
Per cent MoO_3	[a]10.01+	34.98	11.45	.25	[a]6.42+	[a]34.57+	[a]8.80+	[a].31+
Weight MoO_3, pounds	1.802	1.592	.180	.030	4.976	4.432	.355	.189
Extraction MoO_3, per cent		88.35	9.99			89.07	7.13	
Loss MoO_3, per cent				1.66				3.80

[a] Calculated.

Concentration tests, on machines of various types, of wulfenite ore from the Old Yuma mine 14 miles northwest of Tucson, Ariz.—Continued.

TEST 2. CONCENTRATION ON PNEUMATIC PULSATOR JIG.

Item.	Through 20-mesh, on 60-mesh.			Through 60-mesh, on 100-mesh.			Total.		
	Ore.	Concentrates.	Tailings.	Ore.	Concentrates.	Tailings.	Ore.	Concentrates.	Tailings.
Weight, pounds	21.00	4.05	16.95	7.00	0.80	6.20	28.00	4.85	23.15
Per cent MoO_3	[a]7.60+	33.48	1.42	[a]3.94+	29.23	.67	[a]6.68+	[a]32.78+	[a]1.22+
Weight MoO_3, pounds	1.597	1.356	.241	.276	.234	.042	1.873	1.590	.283
Extraction MoO_3, per cent		84.91			84.78			84.89	
Loss MoO_3, per cent			15.09			15.22			15.11

TEST 3. CONCENTRATION ON DIAGONAL-RIFFLE OSCILLATING TABLE (WET).

Item.	Through 100-mesh.		
	Ore.	Concentrates.	Tailings.
Weight, pounds	11.50	0.78	10.72
Per cent MoO_3	[a]2.33+	21.98	.91
Weight MoO_3, pounds	.269	.171	.098
Extraction MoO_3, per cent		63.57	
Loss MoO_3, per cent			36.43

[a] Calculated.

SUMMARY.

Item.	Ore.	Concentrates.	Middlings.	Tailings.
Total weight, pounds	117.00	18.45	4.03	94.52
Total weight MoO_3, pounds	7.118	6.193	.355	.570
Per cent MoO_3	[a]6.08+	[a]33.56+	[a]8.80+	[a].60+
Extraction MoO_3, per cent		87.00	4.99	
Loss MoO_3, per cent				8.01

[a] Calculated.

Distribution of wulfenite, and recoveries and losses, according to sizes in tests 1, 2, and 3.

Machine used.	Size of material.	Per cent of total MoO_3 in—			
		Feed.	Concentrates.	Middlings.	Tailings.
Hydraulic pulsator jig	Through 3-mesh, on 6-mesh	20.98	18.12	1.74	1.12
	Through 6-mesh, on 10-mesh	23.60	21.77	.72	1.11
	Through 10-mesh, on 20-mesh	25.32	22.37	2.53	.42
	Through 3-mesh, on 20-mesh	69.90	62.26	4.99	2.65
Pneumatic pulsator jig	Through 20-mesh, on 60-mesh.	22.44	19.05		3.39
	Through 60-mesh, on 100-mesh.	3.88	3.29		.59
	Through 20-mesh, on 100-mesh.	26.32	22.34		3.98
Concentrating table	Through 100-mesh	3.78	2.40		1.38
Total		100.00	87.00	4.99	8.01

DISCUSSION OF RESULTS OF TESTS.

The results obtained by treatment on the hydraulic pulsator jig show that the concentrates obtained averaged 34.57 per cent molybdic trioxide (MoO_3), which corresponds to 88.12 per cent pure wulfenite, and that the extraction in these concentrates was 89.07 per cent. The total middlings, which amounted to only 4.03 pounds, or 5.2 per cent of the feed, accounted for 7.13 per cent of the remaining molybdenum, and the bulk of the wulfenite contained in them could without doubt have been recovered as a high-grade concentrate by recrushing and treating the product on concentrating tables, even though the middlings contained considerable cerussite. It will be noted that the tailings averaged only 0.31 per cent molybdic trioxide, and although they amounted to 78.26 per cent by weight of the feed, the loss of wulfenite in them was only 3.8 per cent of the total quantity in the feed. In this instance, the ore through 10-mesh and on 20-mesh was jigged only to show that the method was applicable to treating material of this fineness. In commercial work it would probably be preferable to treat this size of wulfenite ore on concentrating tables, as in general more expert attention is required in the successful operation of jigs than in the use of tables.

The tests on the air pulsator jig were made to indicate the applicability of pneumatic processes to wulfenite ores in deposits situated at such distances from water supplies as to make wet concentration out of the question. A summary of the tests shows that the concentrates averaged 32.78 per cent molybdic trioxide, corresponding to 83.56 per cent pure wulfenite, and that the extraction in the concentrates was 84.89 per cent. No middling products were made, and for that reason the tailings were richer in wulfenite than they otherwise would have been. At the end of the two runs a perfect separation of the concentrates and tailings remaining in the jig was impossible, and in the effort to keep the concentrates clean a small quantity of them was skimmed off with the tailings. This procedure also lowered the extraction. Further, closer sizing of the material before treatment would, without doubt, have been beneficial. However, the tests demonstrated that an excellent grade of concentrates can be made by a pneumatic process, and considering all the conditions, the average extraction of 84.89 per cent compares favorably with the average extraction of 89.07 per cent made from the coarser sizes of material by the hydraulic pulsator jig, and it is probably little less than that which would have been obtained if the material had been treated by wet concentration on tables.

A common make of oscillating table with riffles terminating in a diagonal line across the deck was used for the wet concentration of the material that passed through a 100-mesh screen. A good marketable grade of concentrates was made, but the extraction (63.57 per cent) was poor. The results indicate that a considerable porportion of the material was too fine to be treated efficiently on this type of machine and that the ore should be further sized or classified and the finer part treated on a slime table.

Summarizing the results of the three series of tests, it will be seen that the average molybdic trioxide content of the total concentrates, total middlings, and total tailings was 33.56 per cent, 8.80 per cent, and 0.60 per cent, and that the extraction in the concentrate was 87 per cent, and in the middlings 4.99 per cent, whereas the total loss in the tailings was 8.01 per cent.

The table giving the distribution of wulfenite and the recoveries and losses in the various sizes of material, shows that practically 70 per cent of the total wulfenite in the ore was contained in the material through 3-mesh and on 20-mesh, and that out of this amount 62.26 per cent was recovered in the concentrates from the hydraulic pulsator jig, whereas 4.99 per cent more was contained in the middlings from the jig, a total recovery of 67.25 per cent. The table further shows that out of 26.32 per cent of the total wulfenite in the ore contained in the material through 20-mesh and on 100-mesh, the pneumatic pulsator jig recovered 22.34 per cent. Only 3.78 per cent of the wulfenite content of the ore was finer than 100-mesh, and of this amount the concentrating tables recovered 2.4 per cent.

Although the tests discussed above were not conducted with the idea of deriving a method of treatment, the results indicate in a general way the procedure that should be adopted. In the author's opinion the ore could be treated efficiently by wet concentration approximately as follows:

After the ore has been crushed in a jaw or crusher in gyratory crushers to about ½-inch size it should be further crushed in rolls to about ¼-inch and the product screened into three series of sizes. Approximately, these sizes should be as follows: Through 4-mesh and on 8-mesh, and through 8-mesh and on 12-mesh, these two sizes to be treated on jigs; through 12-mesh and on 20-mesh, through 20-mesh and on 40-mesh, through 40-mesh and on 80-mesh, and through 80-mesh and on 120-mesh, these four sizes to be treated on concentrating tables, and the material through 120-mesh to be treated on slime tables. The jigs should be operated so as to obtain as clean concentrates and tailings as possible, and the middlings, after recrushing (perhaps through 20-mesh), should be appropriately sized for reconcentration by the tables.

In the operation of the tables the price received for various grades of wulfenite concentrates would determine where the cut between concentrates and tailings should be made. It might be advanta-

A. TAILING PILES CONTAINING WULFENITE, FROM STAMP AND CYANIDE MILLS, MAMMOTH, ARIZ. LOADING TUNNEL AND TRACK TO WULFENITE MILL IN THE FOREGROUND.

B. BOYKIN & HEREFORD WULFENITE MILL, MAMMOTH, ARIZ.

geous, from a commercial standpoint, to sacrifice something on the recovery in order to make an especially high grade of concentrates.

The author is confident that if the process described above was applied on ore similar to that treated it would result in at least a 90 per cent recovery of the wulfenite as a high-grade concentrate containing 30 per cent or more of molybdic trioxide.

It is of interest to note that the particular lot of ore used in the tests assayed 3.64 ounces of gold per ton and that a microscopic examination showed that much of the gold was contained as native metal in the wulfenite crystals themselves. As far as known, this is the first time that the presence of visible gold has been reported in wulfenite. Further, it was ascertained that the gold was seemingly confined to dark-colored crystals and that the more plentiful orange-colored crystals were free from the element. In order to verify the results of the microscopic examination, two lots of crystals were hand picked from the coarser sizes of concentrates obtained by jigging. One lot consisted of clear, orange-yellow crystals, and the other of dark-brown to almost black crystals. These lots were crushed and assayed. Two assays of the yellow material failed to show even a trace of gold, whereas two assays of the dark crystals yielded 143.32 and 138.02 ounces per ton, thus proving conclusively that the gold was confined to the latter crystals. The author believes that a considerable proportion of the dark color of these crystals is due to minute particles of gold.

It is regretted that owing to the removal of considerable material from the coarser sizes of jigged concentrates for experimental purposes, no statement can be made regarding the recovery of gold in the concentrates as a whole. However, it would probably be somewhat less than the corresponding recovery of wulfenite or of molybdic trioxide, as no doubt the cerussite in the ore contains some gold. For example, the concentrates and tailings derived by jigging the material through 20-mesh on 60-mesh assayed 15.36 and 0.82 ounces of gold per ton. Calculated from these assays, the gold content of the feed was 3.62 ounces per ton, and the recovery in the concentrates was 81.74 per cent, or slightly less than the corresponding recovery of molybdic trioxide, which was 84.91 per cent. The percentage of gold recovered, however, is probably almost identical with that of lead.

BOYKIN & HEREFORD WULFENITE MILL AT MAMMOTH, ARIZ.

At Mammoth, Pinal County, Ariz., there are large piles of tailings (Pl. XVIII, *A*) derived from ore from the Mammoth mine, about 3 miles distant at Schultz.[a] This ore was originally crushed in a stamp mill and treated by amalgamation to recover its free-gold content. Later the tailings from this process were cyanided to extract the considerable part of the gold that was not free milling or had otherwise been lost in the first operation. Subsequently part of the resulting tailings was sluiced to recover the wulfenite content. This last operation was over a decade ago, and it probably supplied about 750 tons of wulfenite concentrates out of the 795 tons of combined wulfenite and molybdenite concentrates that constituted the total production of this county in 1903.[a]

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[a] For description of the ore from this mine see p. 47.

A part of these tailings piles is shown in Plate XV, *B*. They are estimated to contain 200,000 to 250,000 tons of material and 1 to 2 per cent of wulfenite. In 1914 Messrs. F. H. Hereford and R. O. Boykin, of Tucson, Ariz., who had obtained control of all but about 25,000 tons of this material, installed two concentrating tables in the mill building of the old cyanide plant and commenced the re-treatment of the tailings to recover the wulfenite. Operations were so successful that early in 1915 they entirely remodeled the small plant and increased the number of concentrating tables to seven. At this time (June, 1915) operations are being conducted as follows:

The tailings are dug from the tailings piles by means of a four-horse scraper and dragged up an incline to a loading platform, where they are dumped onto a coarse grizzly, which removes pieces of board, brush, etc., and allows the tailings to fall through into a car. The scraper used and the loading platform are shown in Plates XV, *B*, and XVIII, *A*. The cars into which the tailings are dumped, each having a capacity of slightly over a ton, are trammed by hand for several hundred feet to the foot of an incline leading to the mill bin. Here each car is hooked onto a cable and drawn up the incline to the upper edge of the bin, about 30 feet above the ground, and its contents dumped into the bin by hand. Each empty car is returned down the incline by gravity and run onto a switch to allow the next loaded car to pass and then trammed back into the tunnel beneath the loading platform to be refilled. Plate XVIII, *B*, shows a general view of the mill and its surroundings. It is understood that the work of scraping the tailings and placing them in the mill bin is let by contract and that the cost is about 14 cents per ton.

DISCUSSION OF FLOW SHEET.

In the following description of the operation of the mill the numbers refer to the flow sheet shown in figure 2. The mill bin or feed hopper, *1*, has the form of an inverted square pyramid truncated parallel with its base, and holds about 40 tons. It is clearly shown in Plate XVIII, *B*. From this hopper the tailings are washed by a small stream of water over an inclined screen, *2*, of about 4-mesh. The oversize on this screen is removed by hand and thrown away, and the undersize is carried through a launder into the mill building, where it discharges into a hindered-settling classifier, *3*. The overflow from this classifier goes to a second hindered-settling classifier, *4*, and the overflow from the second classifier to a third, *5*, and so on through five classifiers. The overflow from the fifth classifier passes into a settling cone, *8*, the overflow from which goes into the tailings sump, *18*. The spigot products from the first three classifiers, *3* to *5*, are treated separately on three Overstrom tables, *9* to *11*. The spigot product from the fourth classifier, *6*, is divided into two parts, one of which goes to a Ludwig table, *12*, and the other to a Card table, *13*. The spigot products from the fifth classifier, *7*, and from the settling cone, *8*, are treated separately on two Card tables, *14* and *15*. Each of the concentrating tables makes a middlings product, which goes to a storage bin, *17*, for re-treatment. It is understood that these middlings are sometimes re-run on one of the concentrating tables, which is temporarily cut out of the mill system for their re-treatment, and

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[a] Pratt, J. H., The steel-hardening metals: Mineral Resources U. S. for 1903, U. S. Geol. Survey, 1904, p. 308.

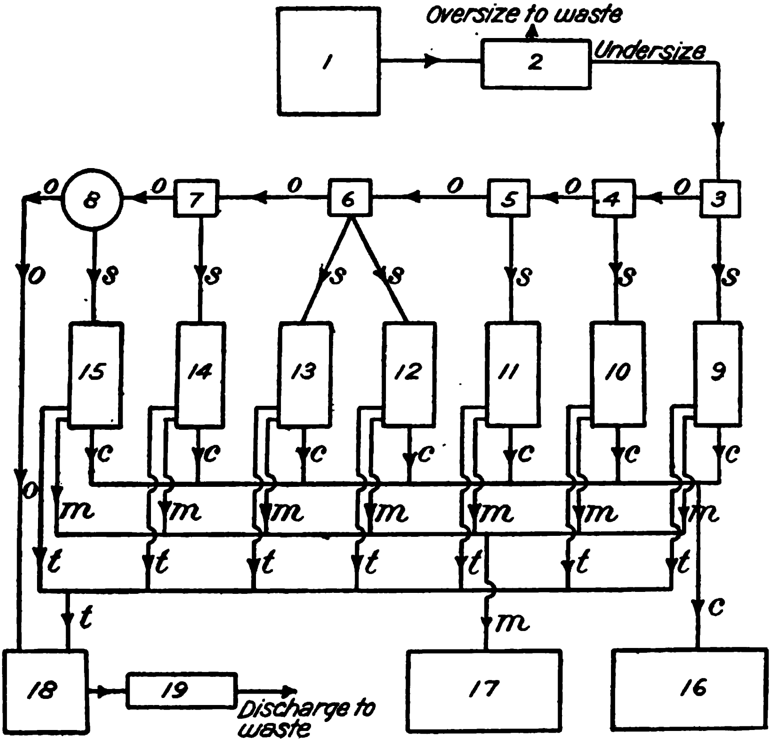

FIGURE 2.—Flow sheet of Boykin & Hereford wulfenite mill, Mammoth, Ariz. *1*, Mill bin or hopper, having a capacity of about 40 tons; *2*, inclined 4-mesh screen; *3* to *7*, hindered-settling classifiers; *8*, settling cone; *9* to *11*, Overstrom tables; *12*, Ludwig table; *13* to *15*, Card tables; *16*, concentrate storage; *17*, middlings storage; *18*, tailings sump; *19*, Byron Jackson 3-inch "Dreadnaught" sand pump.

sometimes they are returned to the mill system as a whole. The tailings from all the tables go to the tailings sump *18*, and the concentrates to storage bin *16*, from which they are taken to be dried and sacked. The tailings are pumped from the tailings sump *18* by a Byron Jackson 3-inch "Dreadnaught" sand pump, *19*, and discharged through a pipe onto a tailings pile a short distance from the mill.

Power is furnished by two Fairbanks-Morse oil engines of 12 and 20 horsepower. The smaller engine drives the sand pump *19*, and the larger engine supplies power for operating the concentrating tables *9* to *15*, a Byron Jackson 4-inch pump that supplies the plant with water, and a Browning 2-kilowatt dynamo which is rated to supply 16.6 amperes at 120 volts when run at 1,425 revolutions per minute. The dynamo furnishes electricity for lighting the mill and for illuminating the scraping and tramming operations on the tailings piles at night. The mill operates 24 hours a day and employs 10 to 12 men.

It is reported that about 2 tons of concentrates are obtained each day from the treatment of about 200 tons of tailings. A large sample of these concentrates, furnished to the author through the courtesy of Messrs. Hereford and Boykin, upon analysis yielded the results shown in the following table:

Analysis of wulfenite concentrates from mill at Mammoth, Ariz.[a]

Constituent.	Per cent.
MoO_3	22.62
PbO	61.83
V_2O_5	1.28
Fe_2O_3 and Al_2O_3	5.50
CuO	.65
SiO_2	1.53
P_2O_5	.30
SO_3	1.96
CO_2	3.20
Cl	.23
CaO, K_2O, etc	[b] .90
Total	100.00

The above analysis indicates the presence of the four principal mineral ingredients of the concentrates in the following percentages; Wulfenite, 57.63; cerussite, 18.32; vanadinite, 8.65; anglesite, 6.52; total, 91.12 per cent. As the last three minerals are of nearly the same specific gravity as wulfenite, the rather low molybdic trioxide content of the concentrates (22.62 per cent) is not surprising. In fact, with such relatively large percentages of these other heavy lead minerals in the feed, the writer does not see how any considerable betterment of the grade of concentrates could be expected. Inasmuch as over 1 per cent of tungsten had been reported to Messrs. Hereford and Boykin in the analysis of a sample said to have represented a considerable tonnage of concentrates, it is of interest to note that none of that element was found in the analysis given above.

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[a] Analyst, H. A. Doerner, Bureau of Mines.

[b] Approximate.

Although it would be difficult to improve greatly the grade of concentrates being made at this plant, the author is of the opinion that the capacity of a plant of this character could be greatly increased by the installation of an efficient screening system to remove the coarser particles of gangue before concentration on the tables, and that the extraction of wulfenite could be considerably improved by the treatment of the fines on slime tables or slime vanners.

RESULTS OF SCREEN TEST.

A screen test of a sample of concentrates was made, the results being presented below. The test showed that 93.6 per cent would pass through a 100-mesh screen, and that 76.2 per cent would pass through a 200-mesh screen. Further, it is reasonable to assume that the losses of wulfenite occurring in the mill are confined almost entirely to very fine material, so the relative proportion of fine to coarse wulfenite in the feed is probably even greater than indicated by the results of the screen test. It is obvious that, as the tables used for treating the finer sizes of material are not suited for treating slimes, a considerable loss of wulfenite must occur in their tailings. Moreover, the overflow from the settling cone in the mill is not treated at all but runs directly to the tailings sump. A panning test of this overflow product showed the presence of considerable fine wulfenite which could at least be partly recovered by allowing the overflow to settle in tanks and treating the slimes on slime tables or slime vanners. Further, as stated above, it is the author's opinion that a much better recovery would be effected if the finer part of the material, embracing the spigot product from the settling cone, and even, perhaps, the spigot product from the fifth, or final, classifier now being handled by the tables, were treated on concentrators adapted to handling slimes.

Results of screen test on wulfenite concentrates from mill at Mammoth, Ariz.

[Screen ratio, 1.414.]

Mesh.	Opening of holding screen.		Weight.	Per cent of total weight.	Per cent of cumulative weight.
	Inch.	Millimeter.			
			Grams.		
On 48	0.0116	0.295	41	2.7	2.7
Through 48, on 65	.0082	.208	21	1.4	4.1
Through 65, on 100	.0058	.147	35	2.3	6.4
Through 100, on 150	.0041	.104	79	5.3	11.7
Through 150, on 200	.0029	.074	182	12.1	23.8
Through 200			1,142	76.2	100.0
Total			1,500	100.0	

The results of the screen test showed that only 2.7 per cent of the concentrates were caught on a 48-mesh screen. In other words, the original feed to the mill might be screened through a 48-mesh sieve, and by throwing away the oversize only 2.7 per cent of the material now recovered would be lost. It is regretted that the results of a screen test on the present feed to the mill are not available, so that the exact proportion of the gangue that might be eliminated by screening with a practically negligible loss might be accurately stated. The writer, however, is confident that more than one-quarter, and perhaps even one-half, of the total feed to the mill could be removed by screening to 40-mesh. That the capacity of the mill would be considerably increased by such a procedure is obvious, and the recovery and the grade of concentrates would probably also be benefited. In fairness to the operators of the mill it must be said that the screening system at the time of the author's visit (screening to about 4-mesh at the outlet of the mill bin and to about 8-mesh or 10-mesh through pieces of screen placed in the feed boxes of the concentrating tables), was only a temporary makeshift, and has doubtless been replaced by a more efficient system.

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Bulletin 112

DEPARTMENT OF THE INTERIOR
FRANKLIN K. LANE, SECRETARY
BUREAU OF MINES
VAN. H. MANNING, DIRECTOR

MINING AND PREPARING DOMESTIC GRAPHITE FOR CRUCIBLE USE

BY

GEORGE D. DUB and FREDERICK G. MOSES

WITH A

CHAPTER ON METHODS OF ANALYSIS USED BY THE BUREAU OF MINES

BY

G. B. TAYLOR and W. A. SELVIG

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MINING AND PREPARING DOMESTIC GRAPHITE FOR CRUCIBLE USE.

By George D. Dub and Frederick G. Moses.

PREFATORY STATEMENT.

The Bureau of Mines, in connection with the investigations of war minerals it conducted, examined the graphite deposits of this country, studied the methods of mining and preparation used, and sought to devise more efficient methods for the preparation of domestic graphite, and to standardize the quality of the product. The graphite investigations covered three general phases, as follows:

1. An examination of the deposits in Alabama and other States, and a survey of the methods of mining and preparation used. In this connection methods of sampling and analysis were noted and experiments made to determine a standard method of sampling and a rapid but accurate method of analysis.
2. Experimental work on the concentration and refining of domestic crucible graphite to improve the quality of the product and to lessen waste.
3. Experimental work in crucible manufacture to determine the properties of domestic flake and the maximum proportions that might be used without impairing the quality of the crucibles. This work was accompanied by photomicrographic study of crucible structure.

In this bulletin are presented the results obtained in the first two phases of the work outlined above. The bulletin is in two parts. The first part describes the methods of mining and milling used, suggests a standard method of sampling finished graphite, and describes a rapid and convenient method of analysis developed at the Pittsburgh station of the Bureau of Mines, to which had been assigned the analytical and microscopic work. The second part describes experiments on the concentration and milling of graphite, which were made at the Salt Lake City, Utah, station. As soon as the reports incorporated in this bulletin were ready, mimeograph copies were distributed to those persons most interested in order to make the results immediately available.

The experimental work on crucible manufacture, assigned to the Columbus, Ohio, station, yielded results which were made known to the industry. A report on this work will be published later as a separate bulletin.

MINING AND MILLING METHODS IN ALABAMA.

By George D. Dub.

INTRODUCTION.

The writer, in connection with the graphite investigation of the Bureau of Mines, war minerals investigations, was charged with the examination of mining and milling methods. During the course of the field work, Alabama, New York, Pennsylvania, and Texas, the principal producing districts in the United States, were visited. In addition, the writer visited a plant in which graphite is refined from "kish," a by-product obtained from iron blast-furnaces and steel plants. The field work was planned to permit of studying the methods of mining and milling and of refining graphite for market. The results of this observation and study are set forth in the following pages. A standard method for sampling finished graphite is suggested, and a rapid, convenient method of analysis used by the bureau is described.

ACKNOWLEDGMENT.

Acknowledgment is made of the whole-hearted cooperation of producers in all of the fields. The Graphite-Producers Association of Ashland, Ala., the members of the staff of the office of the State geologist of New York, and of the various bureaus and boards in Washington have been of great assistance in furthering the aims of this investigation and in making effective various recommendations made from time to time. To H. S. Mudd, mineral technologist of the Bureau of Mines, special credit is due for his painstaking review of the manuscript.

LITERATURE.

A bibliography on graphite has been prepared by Ferguson[a]. The Canadian Bureau of Mines is revising Cirkel's monograph on graphite[b], Prof. B. L. Miller is revising Cirkel's monograph on deposits of Pennsylvania[c] for the State geological survey, and the

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[a] Ferguson, H. G., Graphite in 1917: Mineral Resources, U. S., 1917, U. S. Geological Survey, 1918, pp. 117–119.

[b] Cirkel, Fritz, Graphite; its properties, occurrence, refining and uses: Canada Department of Mines, Mines Branch, 1907, 307 pp.

[c] Miller, B. L., Graphite deposits of Pennsylvania: Topographic and Geol. Survey of Pennsylvania, 1912, 147 pp.

New York State deposits are fully described in a bulletin of the State Museum by H. L. Alling[a]. In view of the detailed geological discussions and the full descriptions of milling and refining apparatus and of methods in the publications mentioned, such phases receive only incidental mention in this report. No statistics except those not available in State or United States Geological Survey bulletins have been incorporated. The aim is simply to describe present practice and recent developments.

DEVELOPMENT OF GRAPHITE INDUSTRY IN THE UNITED STATES.

Before 1915 nearly all the graphite used in crucible manufacture in this country was imported. Little domestic graphite was used in crucibles, that mined being used chiefly for the manufacture of lubricants, paint, foundry facings, and other purposes. In 1915 the demand for graphite crucibles increased greatly, because of the placing of large foreign contracts for munitions and ordnance with American plants, and because of the inferiority of crucibles made from clay other than that from Klingenburg, Bavaria, which had been cut off by the blockade. Certain crucible manufacturers, fearing a shortage of foreign graphite, offered high prices for domestic flake graphite. This stimulated development of the graphite-bearing rocks of Alabama. Also, the uncertainty regarding foreign supplies caused crucible manufacturers to use a larger proportion of domestic graphite in their mixtures than they had ever before attempted.

During 1916 and 1917 the amount of graphite imported was about eight times the domestic production. Imported graphite comes mainly from Ceylon, Madagascar, and Korea. The amorphous graphite from Korea can be easily replaced by that from Mexico, or, if necessary, by development of somewhat lower grade deposits in the United States. Madagascar and Ceylon have furnished about 70 per cent of the total graphite imported, and of this amount approximately 90 per cent has been used in crucible manufacture.

Until the United States entered the war in 1917, the use of Ceylon and Madagascar graphites by American manufacturers was unrestricted as long as guaranties could be furnished to the British and French Governments that the products into which these graphites entered would not fall into the hands of the enemy. On the declaration of war in April, 1917, these guaranties became unnecessary, as they were superseded by broader American defense measures.

During the interval in the winter of 1917–18 when freight conditions were most congested, an embargo against the shipment of domestic graphite was ordered and permitted to remain in force until the early part of March, 1918. The result of this embargo was a stagnation of the domestic mining industry. The removal of the freight restriction on domestic graphite was followed in April by a complete embargo on importations of overseas graphite during the interval April 15, 1918, to July 1, 1918. For the remainder of the calendar year 5,000 long tons was to have been permitted to enter this country. This order was modified to the extent of allowing importations during the period April 15, 1918, to July 1, 1918; the total amount of these imports, however, was to have been deducted from the 5,000 tons scheduled to enter during the last six months of the year.

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[a] Alling, H. L., The Adirondack Graphite Deposits, N. Y. State Museum Bull. 199, 1917, 150 pp.

Toward the end of June, 1918, in view of the necessity for conserving shipping for the direct military program and after stocks of overseas graphite in the hands of crucible makers, refiners, and dealers were found sufficient to last about six months, it was decided after July 2, 1918, to restrict completely the importation of overseas graphite for the rest of 1918. This order was followed on August 10, 1918, by a request from the War Industries Board that all crucible makers use 20 per cent domestic flake graphite in their crucible graphite mixtures for the rest of 1918 with an increase of 25 per cent for 1919. This request carried with it the statement that applications for import licenses of manufacturers not complying with the provisions of the request would not be approved by the War Industries Board.

The War Trade Board ruling of July 2, 1918, was superseded on October 17, 1918, by a ruling permitting imports of overseas graphite by manufacturers whose applications had been approved by the War Industries Board. The purpose of this ruling was to permit the carrying of 3 or 4 months' supply of graphite, in view of the length of time required for shipments from Ceylon and Madagascar to reach the factory in which they were to be used.

ESTABLISHMENT OF PERMANENT DOMESTIC INDUSTRY.

Before the declaration of war in 1914, all crucible makers in the United States, without exception, used clay imported from Klingenburg, Bavaria, in the manufacture of crucibles. Little work of any kind had been done with domestic or English clay. With the declaration of war the cutting off of Bavarian clay forced the crucible maker to turn his attentions to other clays. Although at first serious difficulties were encountered, the clay problem is fairly well in hand with the result that crucible efficiency compares favorably in service with prewar standards. After clay difficulties had been largely surmounted, the problems of crucible manufacture were considered solved. In the course of experiments, no great success attended the use of more than 25 per cent domestic flake graphite in crucible graphite mixtures. It was, therefore, fitting that the Bureau of Mines should thoroughly investigate not only the use of domestic flake in crucible manufacture, but also the methods of manufacturing crucibles from such graphite.

The future of the graphite industry and the effect of improvements and changes in the brass and steel industries have been well outlined by Ferguson.[a] If new and increasing uses can be developed for graphite, it may be possible through the manufacture of graphite articles of commerce in the various mining districts to establish the domestic mining industry on a firmer basis than is possible with many small operating units that produce only crucible stock. The by-products, No. 2 flake and graphite dust, which are drugs on the present market, would then be of value. The graphite-mining industry made a hearty response to the market for crucible grades created by the request of the War Industries Board, and it would be unfortunate if, with the reestablishment of peace, this industry should revert to its prewar basis.

GRAPHITE INDUSTRY IN ALABAMA.

PLANTS.

There are 39 plants in the three graphite-producing counties of Alabama: Clay, Coosa, and Chilton. About October 1, 1918, four of these plants were in course of erection and fourteen were temporarily closed because of changes in method of treatment or because of plant fires. A little over half of the plants were then operating, and only six were running full time. After the cessation of hostilities production fell rapidly, and in June, 1919, probably not more than half a dozen plants were in operation.

As this industry developed chiefly during 1917 and 1918, operating difficulties still occur.

The plants enumerated have actual operating capacities ranging from 4 to 15 tons an hour, but the rated capacities are about double these figures.

ORE MINED AND METHODS OF MINING.

The graphite occurs in the Talladega schist and is accompanied by quartz, feldspar, mica, other accessory minerals, and alteration products. Only the upper 30 to 60 feet of the graphite schist is mined. This consists of decomposed and weathered schist and is normally rather soft and easily broken, while the unweathered schist or

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[a] Ferguson, H. G., Graphite in 1917: Mineral Resources U. S., 1917, U. S. Geol. Survey, 1918, pp. 97–119.

"blue rock" is comparatively hard and contains considerable pyrite. For this reason, operators are loath to work the "blue rock." Alabama ore contains, as far as is known, no amorphous graphite.

The ore is covered with 1 to 6 feet of overburden. This carries some graphite, but contains so much clay and vegetable matter that it is removed and dumped as waste to obviate milling difficulties caused by the presence of these materials. The stripping is done with plows, scrapers, and wheelbarrows, and does not involve much labor or expense.

All of the companies have open-pit workings. Machine drills are employed at only one mine, where self-rotating hammer drills of the jackhammer type are used to drill holes 15 feet in depth. All other drilling is done by means of a "jumper" drill, holes being drilled 8 to 30 feet deep. All blasting is done with black powder. The deeper holes are chambered with dynamite or blasting gelatin.

The broken ore is loaded into cars by hand, or by steam shovels with a dipper capacity of five-eighths of a cubic yard. With hand loading, end-dump or side-dump cars of 1 to 1¾ ton capacity are employed. With steam shovels, side-dump contractors' cars of 4 to 5 ton capacity are used. About one-fourth of the plants in this district are so situated as to necessitate hoisting ore out of the open pit to the mill. At the plants in which the crude ore enters the mill by gravity, hand tramming is employed, except where the open pit is situated at a considerable elevation above the mill; in such plants, boxed chutes with hand tramming or gravity planes are used. Balanced hoisting is used in only three or four plants. At one plant, a narrow-gage railroad transports ore from the open pit to the mill.

The absence of enough storage room at the head of the mill or at some point between the open cut and the mill is practically general in the district. Only a small proportion of the plants have storage capacity sufficient to permit the accumulation of enough ore to run one shift of 10 hours, the majority of plants having storage capacity to enable milling to continue only for a half shift. Mining is carried on in the open and is dependent, to a certain degree, on the weather. Shut-downs in the mills, owing to operating troubles, are frequent. The result, therefore, of the lack of storage is a loss of operating time, the elimination of which would soon pay for the installation of adequate storage. This point can not be too strongly urged for these small, and, mostly, single-unit plants.

The ore in Alabama averages about 2½ per cent graphitic carbon. From this is obtained generally three products—No. 1 crucible flake, No. 2 flake, and dust. The aim in concentrating is to recover as much as possible of the No. 1 flake, analyzing 85 per cent graphitic carbon and remaining on a No. 8 silk cloth of 86 mesh. No. 2 flake, analyzing 75 to 80 per cent graphitic carbon and of finer size than the No. 1 flake, is a by-product grade for which the market is limited. The dust analyzes 30 per cent or more graphitic carbon and is a greater drug on the market than the No. 2 flake. Hence, the problem of ore dressing is complicated by the necessity of recovering as much as possible of the graphite in the ore in such form as to be marketable as No. 1 flake. The measure of efficiency, therefore, in graphite milling is not the percentage of recovery but the number of pounds of No. 1 flake recovered per ton of ore.

Unfortunately graphite plants do not make a practice of analyzing their mill heads. It is impossible therefore, to establish a relation between the total content of graphite in the ore and the amount recovered as No. 1 flake. Average figures as to the actual production per ton of ore are stated on page 27.

The by-product grades can readily be used in the manufacture of lubricating flake, paint stock, foundry facings, stove polish, etc., but the demands of plants manufacturing these products are not large and the market does not absorb all the No. 2 flake and dust produced.

CRUSHING METHODS.

Many different crushing machines and combinations are used in Alabama in the dressing of graphite ore. The coarse crusher most commonly used is a jaw crusher of the Blake or Dodge type that reduces the ore to about 1½ inch size. In some plants, a toothed impact roll is used for coarse crushing. In plants where dry material is crushed fine, rolls, dry mullers, or Symons disk crushers are used to reduce the material to final treatment size which is about $\frac{1}{16}$ or $\frac{3}{32}$ inch. For fine crushing in plants in which an oil-froth flotation method of concentration is used, coarse crushing is followed by some type of rotating mill, ball mill, pebble mill, or rod mill. These machines are sometimes preceded by a Symons disk crusher.

CONCENTRATION METHODS.

There are four methods of concentrating graphite ores, water "skin-flotation," the pneumatic process, the log-washer process, and oil-froth flotation.

The extent to which these various processes are used in Alabama is as follows, the figures including plants that contemplate putting in those systems under which they have been classified: The skin-flotation washer system is used in twelve and one-half plants. One of the plants consists of two units, one of which is the Minerals Separation oil-froth flotation. The pneumatic system is used in three plants, the log-washer system in two plants, and oil-froth flotation systems in twenty-one and one-half plants.

WATER "SKIN FLOTATION."

In the water "skin flotation" system, a typical flow sheet of which is shown in figure 1, the ore is crushed dry to $\frac{1}{16}$ or $\frac{3}{32}$ inch. It is then sent through a rotary drier, screened, and passed over an air separator where it is classified into three products—"scalpings" or

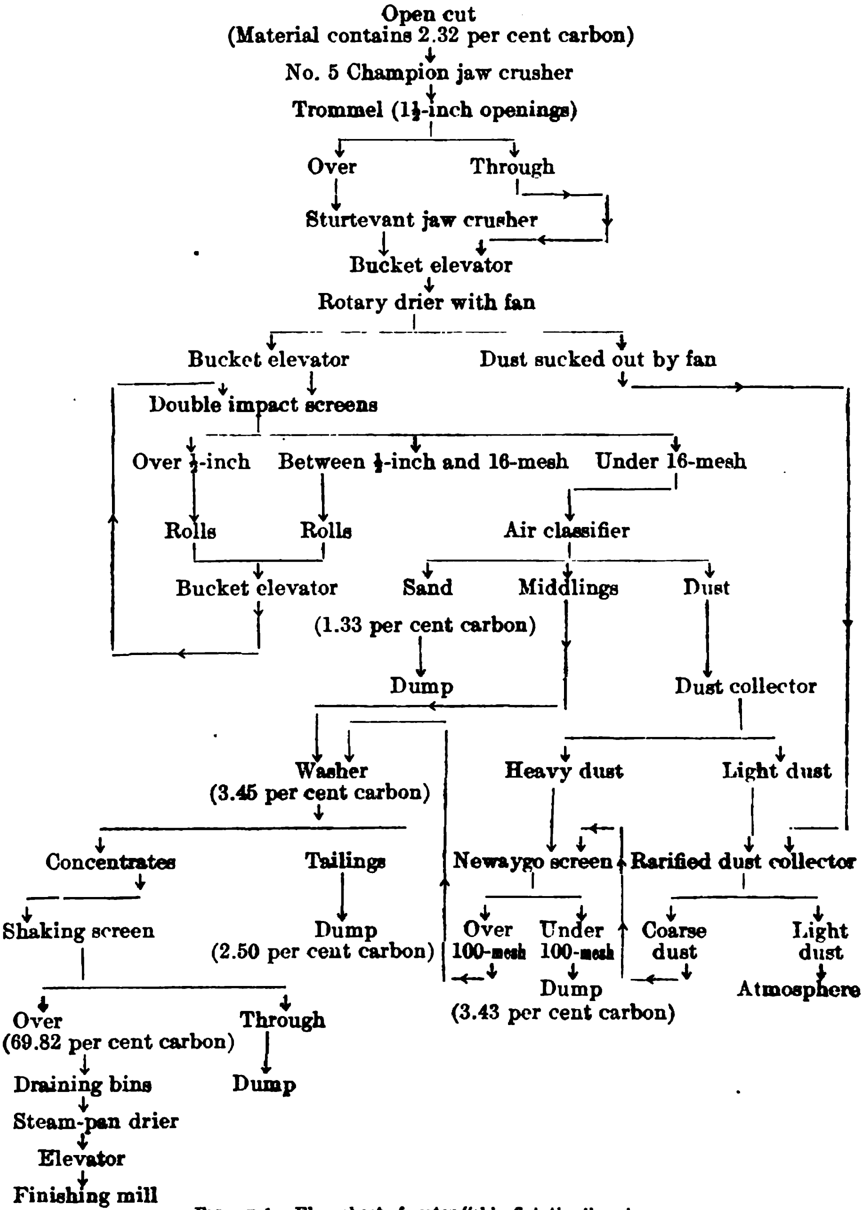

FIGURE 1.—Flow sheet of water "skin flotation" system.

tailings, that go to the waste dump; middlings, that form the "washer" feed; and fines, that are generally treated on a separate group of "washers." The middlings plus the fines, which form 50 to 65 per cent of the crude ore, go to a bin for fine ore, and are fed to "surface-tension" washers where the graphite and some of the mica are floated off, on the surface film of water. The gangue minerals are wet by the water, sink to the hutch of the washer, and are discharged to the tailings launder. The rough graphite concentrate is then dewatered on a trommel or shaking screen, which also removes both the graphite too fine to make No. 1 flake and the fine sand that has been carried over into the concentrates by the film formed by the graphite on the surface of the water. The dewatered concentrate, a dry sample of which analyzes 40 to 60 per cent graphitic carbon, is then dried in a rotary or steam-pan drier and sent to the finishing plant. Average figures for the amount of rough concentrate recovered per ton of ore are not available, but depend of course in large measure upon the grade of ore and of concentrate.

The types of screening and air-classifying apparatus used differ widely, and many of them are unique. Newaygo and "whip-tap" screens are most popular, but other types of impact and shaking screens are used, as well as hexagonal trommels. Most of the air-classifying apparatus is individually designed and erected, and gives results that seem to be efficient. Practically nothing has been done to check up the work of this class of machine; its operation is judged entirely by looking at the products without having even the "scalpings" analyzed. So far as is known, no sufficient quantity of No. 1 flake would be recovered to justify additional treatment, with a view to obtaining more No. 1 flake from these "scalpings."

The result of sampling crude ore, or crude concentrates, and "scalpings" is shown in the table following. All samples were analyzed at the Pittsburgh experiment station of the Bureau of Mines.

Results of operation of air classifier.

[Figures represent percentages of graphitic carbon.]

Heading.	Graphitic carbon, per cent.	Graphitic carbon in "scalpings" or tailings, per cent.
Operating on crude ore:		
First sample	3.45	1.35
Second sample	2.55	1.01
Operating on crude concentrates:		
First sample	62.80	4.68
Second sample	48.80	1.63

There are three types of "skin flotation" washers sufficiently distinct in principle to permit individual description. Figures 2, 3, and 4 illustrate the underlying principles in design. The dimensions given are estimates and not actual measurements.

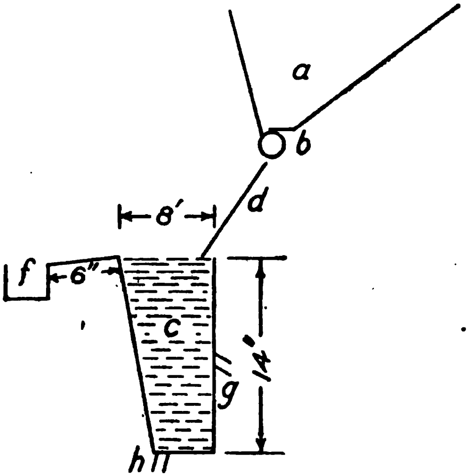

FIGURE 2.—Section through original Munro washer (dimensions indicated are approximate). *a*, Stock bin; *b*, feed cylinder (2-inch revolving shaft); *c*, lake 8 inches wide; *d*, baffle inclined 50° to 65° from horizontal, 10 inches long; *f*, collecting launder, into which graphite is carried over an apron from *c*; *g*, water inlet; *h*, hutch spigot.

MUNRO WASHER.

The Munro washer is a rectangular washer in which the feed drops from the feed cylinder to an inclined flat baffle and thence to the water film that carries the graphite to the collecting launder and permits the gangue to sink to the hutch. These washers are built in units about 4 feet long, placed end to end and back to back.

COLMER WASHER.

The Colmer washer is a circular washer with a conical feed baffle. The ore falls from the baffle on a revolving circular disk with ribs cast on it. The tangential motion thus imparted to the ore is intended to assist in carrying off the graphite by reducing the tendency of the falling particles to rupture the surface film of water. Some operators claim that better results are obtained with this washer by keeping the disk stationary, relying on the disk solely to change the direction of travel of the particles of ore. These operators claim that the tangential motion given by the revolving disk produced a lower-grade concentrate than was obtained by keep-

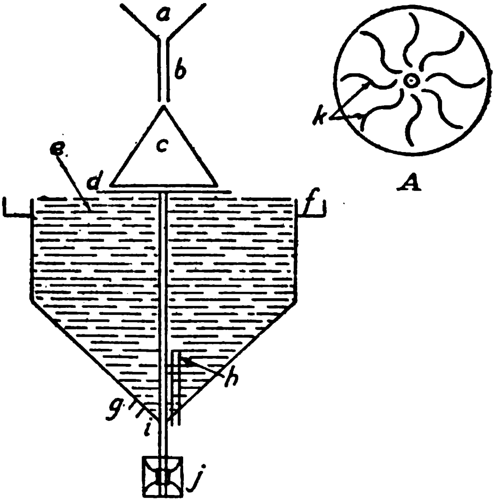

FIGURE 3.—Section through Colmer washer (not drawn to scale). *a*, Ore bin; *b*, feed pipe; *c*, conical feed plate; *d*, revolving disk; *e*, lake about 9 inches wide; *f*, collecting launder; *g*, hutch spigot; *h*, water inlet; *i*, shaft; *j*, pulley for rotating disk; *k*, ribs. A, detail of disk *d* (about 18 inches in diameter).

ing the disk stationary. As shown in figure 3, the washer is about 42 inches in diameter.

NEW MUNRO WASHER.

The new Munro washer (fig. 4), which also is circular in shape, is 6 to 12 inches larger in diameter than the Colmer, most of this increase in size being taken up by increasing the width of the "lake." In the Munro washer, the treatment water is forced through an annular opening, formed by placing a cast-iron cover over a bell-mouth casting which is attached to the feed-water pipe. This washer does away with the mechanical motion of the Colmer type. It is difficult, however, to keep an unobstructed flow of water through the annular opening.

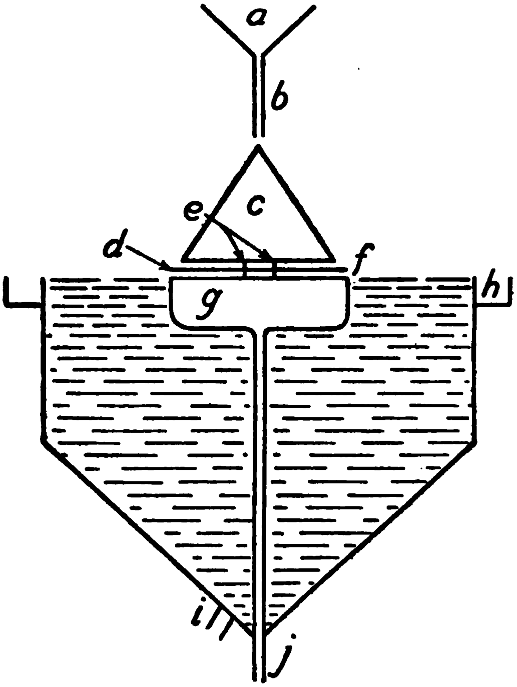

FIGURE 4.—Section of new Munro washer (not drawn to scale; lake about 12 inches wide). *a*, Stock bin; *b*, feed pipe; *c*, conical feed plate; *d*, cover plate; *e*, set screws for regulating width of opening; *f*, annular opening; *g*, bell mouth; *h*, collecting launder; *i*, hutch spigot; *j*, feed-water pipe.

PNEUMATIC PROCESS.

In the treatment of graphite ores by the pneumatic process, pneumatic apparatus is used throughout. A typical flow sheet for this system is not given, because the system has been successful in only one mill. The details of the operation of this plant can not be made public.

LOG-WASHER PROCESS.

A typical flow sheet of the log-washer process is shown in figure 5. In this method of treatment the crude ore is crushed and screened without drying. It is then treated in log washers, kerosene oil being added to the water in the apparatus. The concentrates are screened, washed on a cement floor, drained, and dried. This system eliminates the expensive preliminary drying of the crude ore, necessary with the water "skin flotation."

The simplicity and effectiveness of this plant are clear to the observer. The grade of rough concentrate made is excellent and the only important loss occurs in the coarse-sand tailings, which with regrinding might be made to yield more flake graphite. A test of the coarse-sand tailings, in the same way that has been suggested under the discussion with reference to the "scalpings" of air classifiers, would determine the desirability of regrinding. The fine-sand tailings are remarkably free from detached flakes of graphite.

OIL-FROTH FLOTATION SYSTEMS.

The majority of the graphite plants in Alabama have installed some type of oil-froth flotation cell. Eight plants have installed the Callow pneumatic cells, four have installed the Simplex type of cell, three and one-half (a two-unit plant has one skin-flotation washer system) have installed the Minerals Separation cells, and six plants have installed, or intended to install, homemade cells or washers combining some of the principles of these processes.

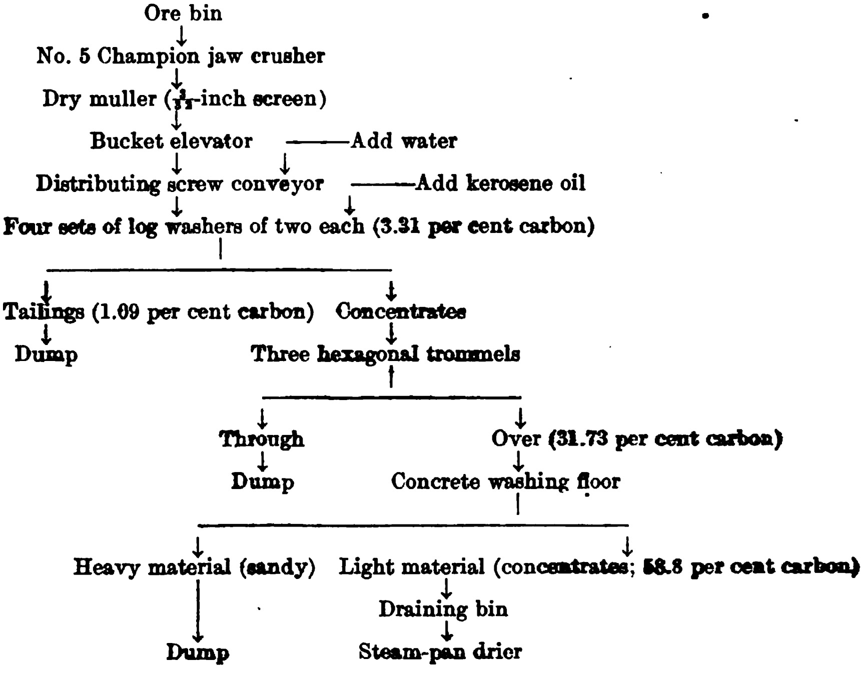
Ore bin
No. 5 Champion jaw crusher
Dry muller (1/32-inch screen)
Bucket elevator
Add water
Distributing screw conveyor
Add kerosene oil
Four sets of log washers of two each (3.31 per cent carbon)
Tailings (1.09 per cent carbon)
Concentrates
Dump
Three hexagonal trommels
Through
Over (31.73 per cent carbon)
Dump
Concrete washing floor
Heavy material (sandy)
Light material (concentrates; 58.8 per cent carbon)
Draining bin
Dump
Steam-pan drier

FIGURE 5.—Flow sheet of log-washer process.

The chief difficulty with oil-froth flotation systems is in the loss of the large flake. In recovering the large flake, so low grade a concentrate is obtained that an added burden is placed on the finishing plant. There is no difficulty in obtaining a high recovery of the high-grade dust, but there is at present no ready market for this character of material. The best results obtained in the district are from the homemade cells or washers mentioned.

In both the Minerals Separation and the Callow systems, fine crushing is accomplished by Marcy, Lehigh, Marathon, or Hardinge ball or pebble mills. In practically all of the plants in which these two systems have been installed, water classifiers of the Deister or Gemmell type are used. Two plants have Dorr classifiers. For dewatering the rough concentrate, vacuum filters of the Portland or Oliver type are generally used rather than shaking screens.

MINERALS SEPARATION SYSTEM.

A typical flow sheet of the Minerals Separation system is shown in figure 6. Assay results for this system are not available.

CALLOW PNEUMATIC SYSTEM.

A typical flow sheet of the Callow pneumatic system is shown in figure 7. This system has received greater development in the Alabama district than any other oil-froth flotation system. At one plant the tailings from the Callow cells are treated in an improvised washer with satisfactory recovery.

Samples obtained in Callow plants did not make a good showing, chiefly because of the physical difficulties in taking them. The results of sampling follow:

Results of operation of Callow pneumatic cells.

[Figures represent percentages of graphitic carbon.]

Test.	Crude ore.	Crude concentrates.	Tailings.
No. 1	2.24	43.66	Sample "salted."
No. 2	Sample lost	52.59	No samples possible.
No. 3	No sample taken [a]	57 to 80	0.5 to 0.9.

[a] At this plant the flake seemingly is being subjected to excessive grinding, with the production of a large quantity of graphite, which will not produce No. 1 flake.

SIMPLEX SYSTEM.

The Simplex system includes crushing as well as concentration. The crushing apparatus has no particular advantage over other crushing machinery but the concentration method appears to be efficient.

In this system, the pulp comes unclassified to the center of an elliptical washer on the surface of which jets of water, with flotation oils, are forced at a pressure as high as 40 pounds per square inch. The jet of water entrains air, and a froth is formed which floats the graphite over the side of the washer, while the gangue drops to the hutch.

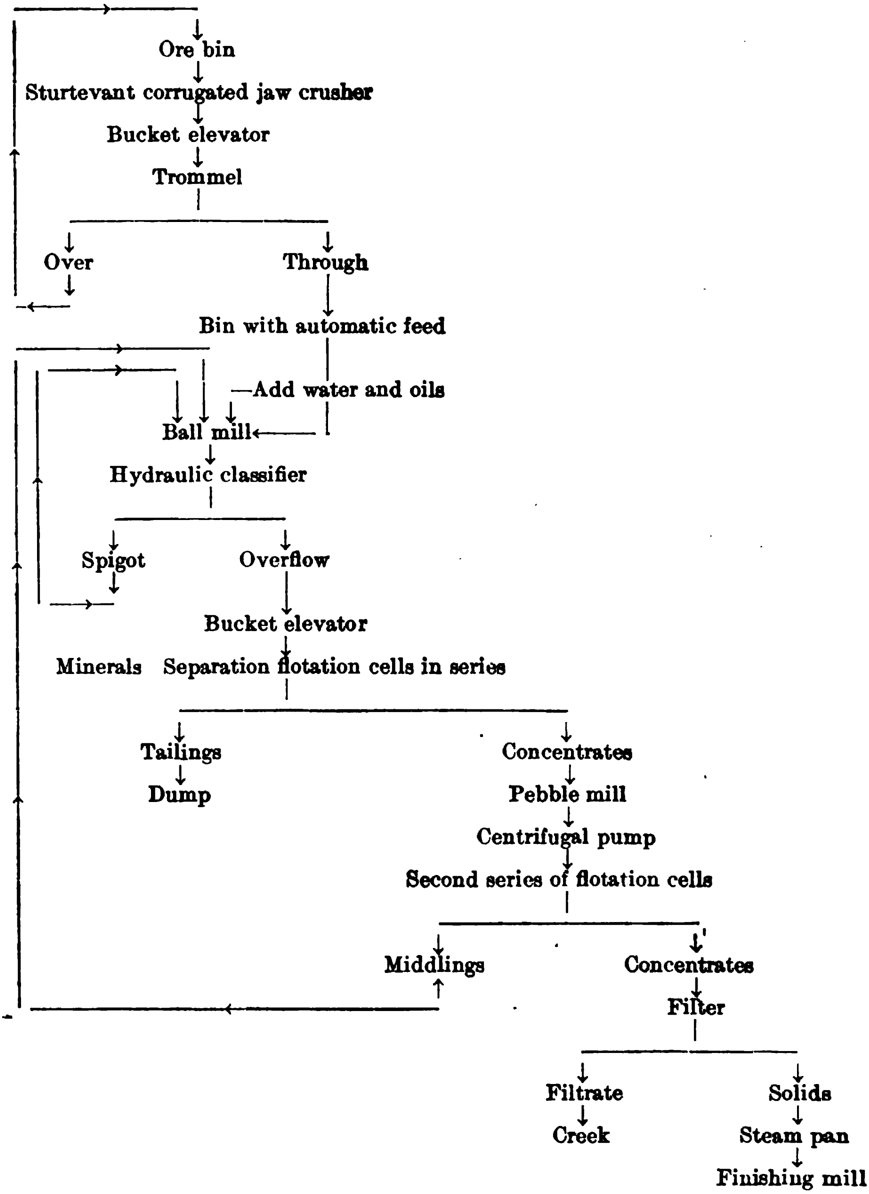

FIGURE 6.—Flow sheet of Minerals Separation system.

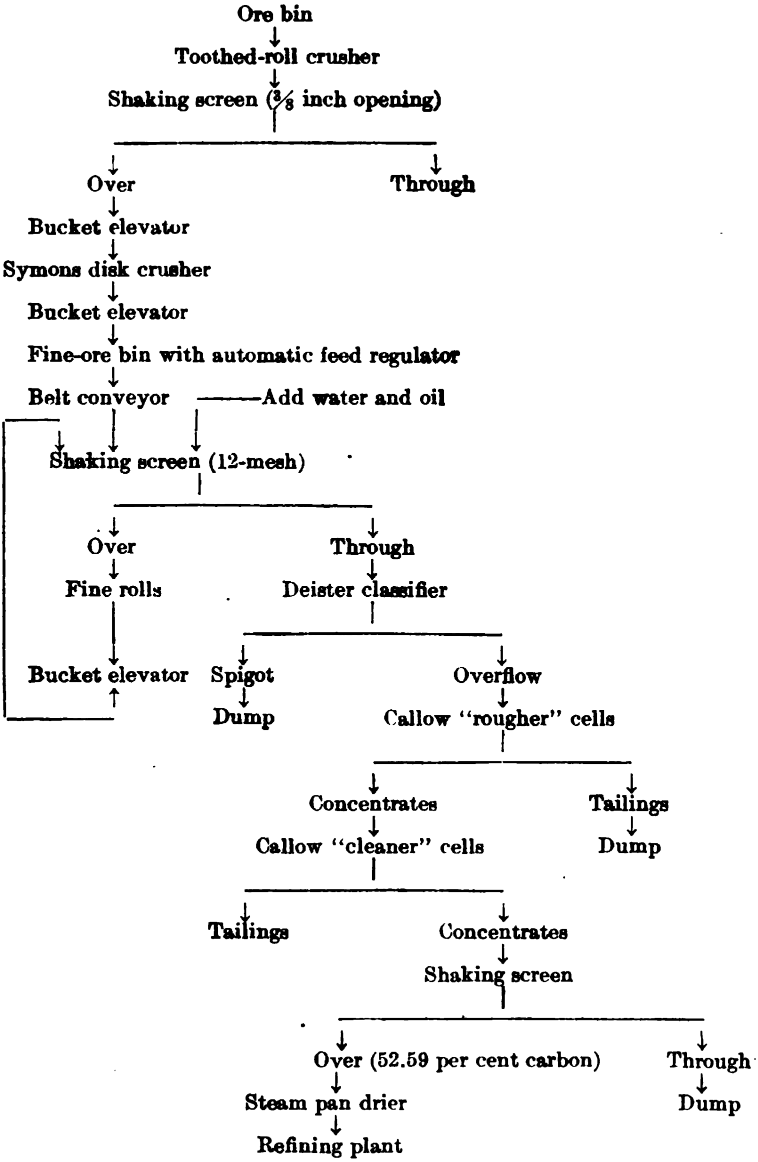

FIGURE 7.—Flow sheet of Callow pneumatic system.

A typical flow sheet for a plant of this type is shown in figure 8, and a sketch of a section through the washer is shown in figure 9.

In various mills in Alabama, Wilfley and Deister tables are being installed to raise the grade of the rough concentrate and to remove gangue from the concentrate fed to the finishing plant. The gangue wears the surfaces of the buhr stones, thus necessitating their more frequent dressing, an operation that is both tedious and expensive.

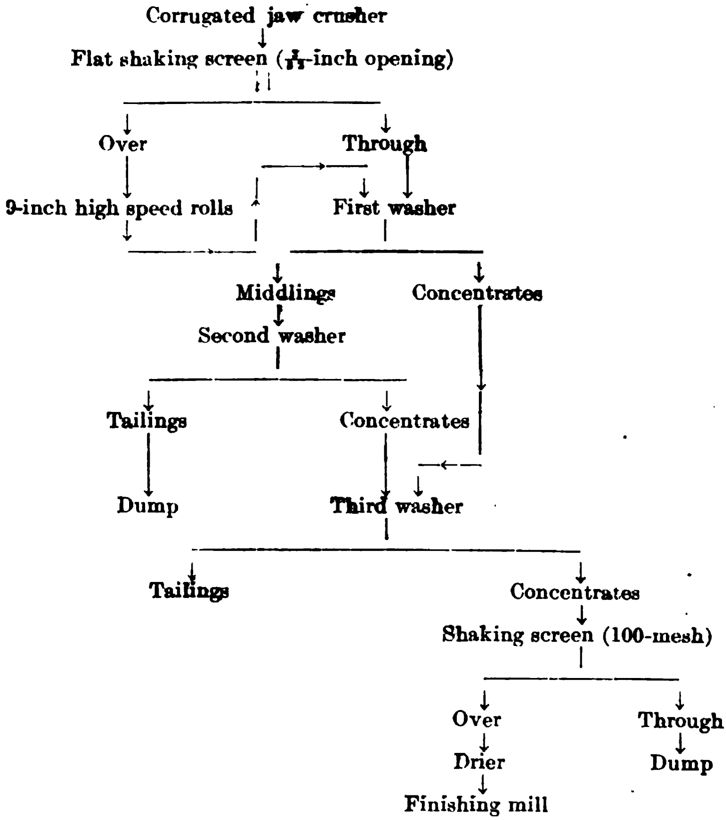

FIGURE 8.—Flow sheet of Simplex system.

At one of the plants, concentrates from a table are treated in an improvised pebble mill. The dust is then removed, and the material is passed through a series of screens, producing No. 1 flake ready for market.

The most important consideration as regards the use of concentrating tables is their capacity. As the use of tables in this district is still in the experimental stage, reliable data as to the success of this new practice is not available.

REFINING METHODS.

The rough concentrate as it comes from the concentrating plant contains a minimum of 40 per cent graphitic carbon. In some plants, this crude concentrate analyzes as high as 75 per cent graphitic carbon, although the usual proportion is 40 to 60 per cent. The impurities are quartz, mica, wood fiber, etc. Material of this character is not readily marketable except at prices far below those obtained for No. 1 flake; moreover, the market for crude concentrate is limited. These conditions, therefore, necessitate the refining of crude concentrate to No. 1 flake, containing 85 per cent graphitic carbon, and remaining on a No. 8 silk cloth of about 86 mesh.

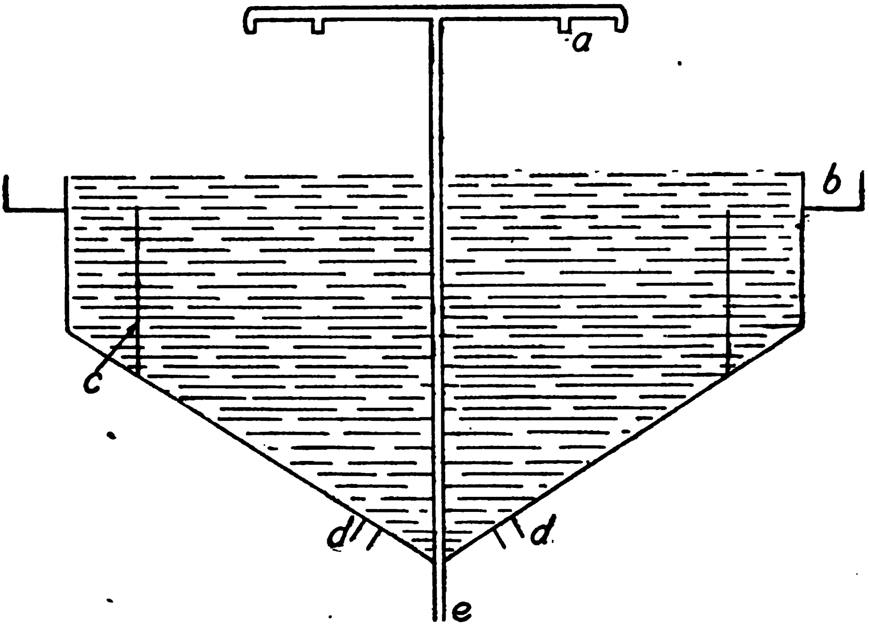

FIGURE 9.—Section of Simplex washer (not drawn to scale; major axis is about 6 feet and minor axis about 3½ feet). *a*, Spigots, ¼ to ⅜ inch orifice, water and oil under pressure; *b*, collecting launder; *c*, screen baffle; *d*, hutch spigots; *e*, feed water.

Microscopic examination shows that the graphite flakes consist of thin laminae of graphite and that present between the laminae are associated minerals, chiefly quartz and mica, which must be eliminated. There are two general methods of refining:

(1) If the crude concentrate comes to the finishing plant with most of the impurities still interlaminated with or attached to the graphite, grinding in a pebble or buhr mill is necessary in order to loosen the graphite from the impurities.

(2) On the other hand, if the crude concentrate comes to the finishing plant with the larger part of impurities as detached par-

ticles, refining can be done pneumatically or with electrostatic machines. Where this method is employed, crushing in the concentrating plant has been of such a character as to accomplish what is usually done in the finishing plant with the buhr or pebble mill.

The first method is the one most generally used; the second method is used in only two plants, and its application is not broad enough to warrant more detailed description at this time.

A flow sheet of a refining plant using the first method is shown in figure 10. This flow sheet is not typical because most plants are quite dissimilar, but it represents in a general way the underlying principles governing refining.

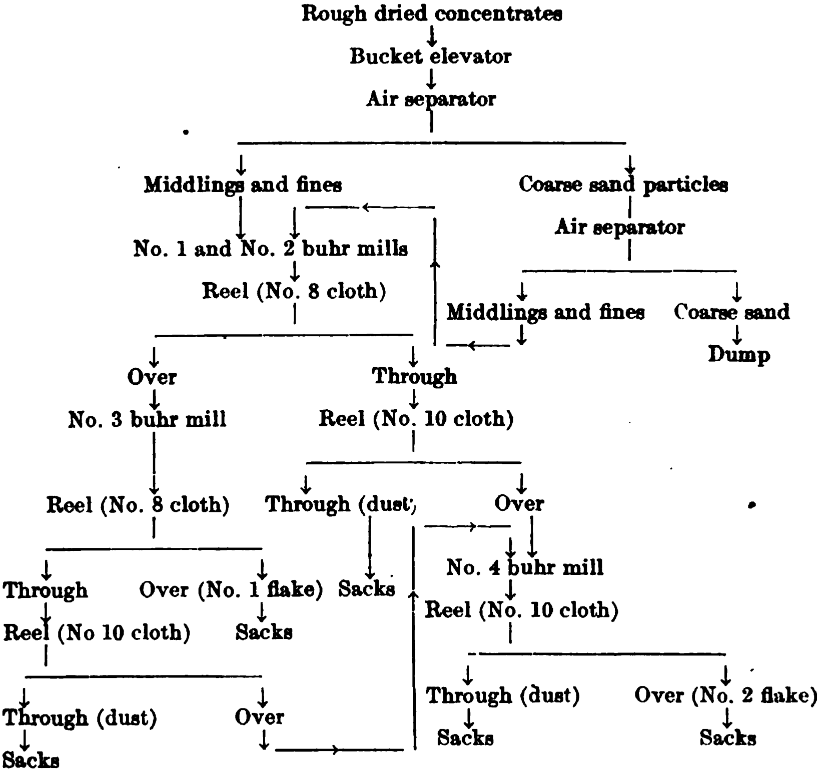

FIGURE 10.—Flow sheet of graphite refining plant.

AIR CLASSIFICATION.

Air classification is used in most of the refining plants and forms an important step in the refining process. Figure 11 shows sketches of air classifiers. Such apparatus is usually improvised. So far as is known no experimental work has been done by the large majority of operators with a view to determining the most efficient proportions and shape for this device.

In principle air suction created by a fan at the back end of each chamber is applied to a falling stream of graphite ore or concentrate. The heavier material, quartz grains with attached graphite, is practically unaffected by the air suction, and falls into the hopper farthest from the fan. The lighter particles are drawn nearest the fan, which sucks the finest dust into the atmosphere. When this apparatus is used on crude concentrates, the middlings—which include the heaviest flake—form No. 1 flake stock, going to the No. 1 buhr mills; the finer material nearest to the fan is screened to remove as much finished No. 1 flake as possible.

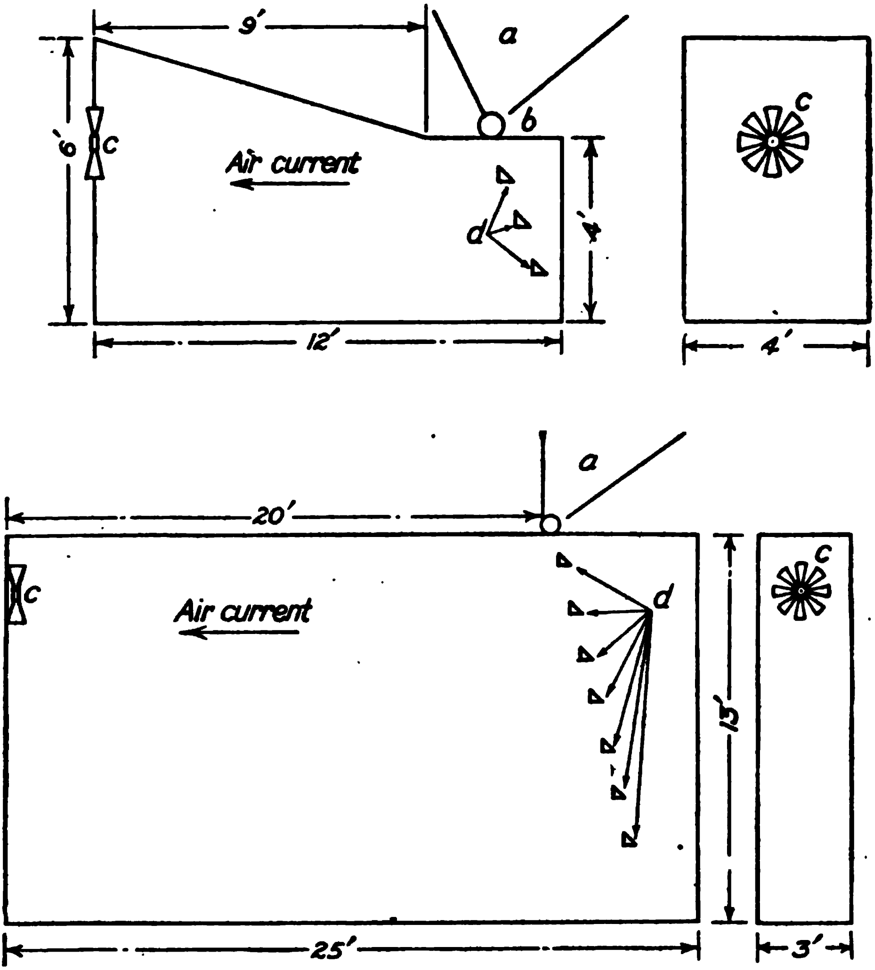

FIGURE 11.—Two types of air classifiers (not drawn to scale). *a*, Stock bin; *b*, feed shaft; *c*, fan; *d*, baffles. Three sets of hoppers are provided for the three products usually made.

In all refining methods the following steps are taken to facilitate the highest possible recovery of No. 1 flake:

1. All dust is eliminated from the circuit as soon as practicable.

2. No. 1 flake is removed from the system as soon as it has been made.

3. As little concentrate as possible is sent to the grinding apparatus.

For analyses of products see discussion of air classification under "Concentration Methods," page 15.

RECOVERY OF FINISHED PRODUCTS.

In discussing recovery in graphite plants it should be pointed out that the individual plants are small mills in which systematic sampling and weighing of products are not regularly employed. No data, therefore, are available as to the extraction obtained at various stages of concentrating and refining. It is impossible, also, to state where the greatest losses occur in these operations except from observation and rough sampling in the field. The results of this sampling are indicated wherever practicable on the flow sheets presented herein; the points at which the greatest losses occur have also been indicated.

The only products actually weighed and sampled are the finished products. It would be entirely feasible to sample regularly the crude ore, the tailings from the roughing mill, and the crude concentrates. Various other products could be sampled from time to time, and this work would amply repay the trouble of sampling.

Recoveries of finished materials are as follows:

1. No. 1 flake analyzing 85 to 90 per cent graphitic carbon and remaining on a No. 8 (86-mesh) or a No. 12 (125-mesh) silk cloth—the smaller flake being obtained when a 90 per cent graphite is produced: 10 to 30 pounds is obtained per ton of ore. The average is about 19 pounds.

2. No. 2 flake, analyzing 75 to 80 per cent graphitic carbon and passing through the meshes above mentioned: 2 to 15 pounds is obtained per ton of ore. The average is about 5 pounds.

3. Dust, analyzing 30 per cent or more in graphitic carbon: An average of about 3 pounds per ton of ore is obtained.

If there were a market for No. 2 flake and dust, a greater recovery of these materials per ton of ore could be made.

The recovery percentages for all products are shown in the table following, which represents average milling practice in the district:

Data showing average recoveries of graphite products in Alabama mills.

Material.	Carbon.	Weight.	Weight multiplied by per cent.	Recovery.
	Per cent.	*Pounds.*		*Per cent.*
No. 1 flake	87	19	1,653	33.06
No. 2 flake	77	5	385	7.70
Dust	30	3	90	1.80
Total finished products	....	27	2,128	42.56
Tails (by difference)	1.46	1,973	2,872	57.44
Crude ore	2.5	2,000	5,000	100.00

The amount of No. 1 flake produced per man per hour ranges from 4½ to 8 pounds, and averages about 6 pounds, figured on the total number of men and officials employed. Proportionate quantities of No. 2 flake and dust are obtained. There is no definite advantage apparent from figures obtained from the different classes of plants. The outputs, of course, depend to a large extent on the amount of graphite originally present in the ore, so that although these figures are an index of costs, they can not be used as a measure of comparative efficiency.

COMPARATIVE COST OF ERECTING PLANTS.

The cheapest type of plant to install is that using a log washer, and this is probably the cheapest to run. No figures are available as to either initial or operating costs for a plant of this kind.

For a plant capable of treating 10 tons per hour, the initial cost ranges from $35,000 to $60,000 erected, not including that of a finishing plant, which would add $5,000 to $10,000 more to the cost.

FIELD SAMPLING.

In the flow sheets accompanying this report, available assays of samples taken in the field have been inserted, and these give some idea as to the relative efficiency of different methods of milling and of steps in the various processes. In many instances where assays have been omitted, no samples were taken; and sometimes the samples taken were lost either in transit or in drying in the field, or the assay results were inconclusive because of the difficulty of getting a representative sample in the plant.

GRAPHITE INDUSTRY IN NEW YORK.

PLANTS.

The mining of graphite in New York was started about 60 years ago. This State for many years produced more graphite than any other, but since 1915 has yielded first place to Alabama, where the growth of the industry has been remarkably rapid.

In 1918 there were three mines in operation, namely, the mine of the Graphite Products Corporation, 3 miles north of Saratoga Springs; Hooper Bros. mine, 4 miles west of Whitehall; and the mine of the American Graphite Co., worked by the Joseph Dixon Crucible Co., 4 miles west of Hague.

OCCURRENCE OF ORE.

In New York the ore averages 4 to 6 per cent graphitic carbon as compared with 2½ per cent in Alabama. There are two important types of graphite deposits in the Adirondack foothills of New York—those on the contact of a limestone and pegmatite, and those on the contact of the Hague garnet-sillimanite gneiss with the Faxon limestone or Swede Pond gneiss. In deposits of the former type, large flakes of graphite are obtained, but such deposits are extremely pockety, and their development has not been profitable. Present production is from the second type of deposit, in which the ore is quite regular and is of a more uniform grade.

The ore on the whole is much harder and more siliceous than the Alabama ore and resembles in many respects the unweathered "blue rock" of Alabama, the original bluish Talladega schist.

MINING METHODS.

Graphite ore is mined in New York both in open pits and underground. The larger part of the ore comes from underground mining. All drilling is done with power drills. On account of the siliceous character of the ore, a greater quantity of fines is produced during crushing than is obtained in Alabama. The ore-bearing zone is usually not over 25 feet thick and averages about 15 feet.

OPEN-PIT MINING.

Wherever possible, the open-pit system of mining is used because of the ease and cheapness of preparing an open pit, where there is no excessive amount of overburden that requires stripping.

UNDERGROUND MINING.

The deposits dip 25° to 35° so that it soon becomes necessary to go under cover for the ore in the course of the development of any mine. This has its advantage in the North because of the long and severe winters which hamper open-cut work.

The system of underground mining at the largest mine in the district is the room-and-pillar method with underhand stoping. In new developments in the district, a definite system of mining has not as yet been determined.

CONCENTRATING METHODS.

A flow sheet of one of the plants in New York is given in figure 12. In this plant gravity stamps are used as fine crushers and "buddles" as concentrating machines. Concentrating tables are used for treating "buddle" middlings, whereas, in Alabama they are to be employed in raising the grade of rough concentrates preparatory to treatment in the finishing mills.

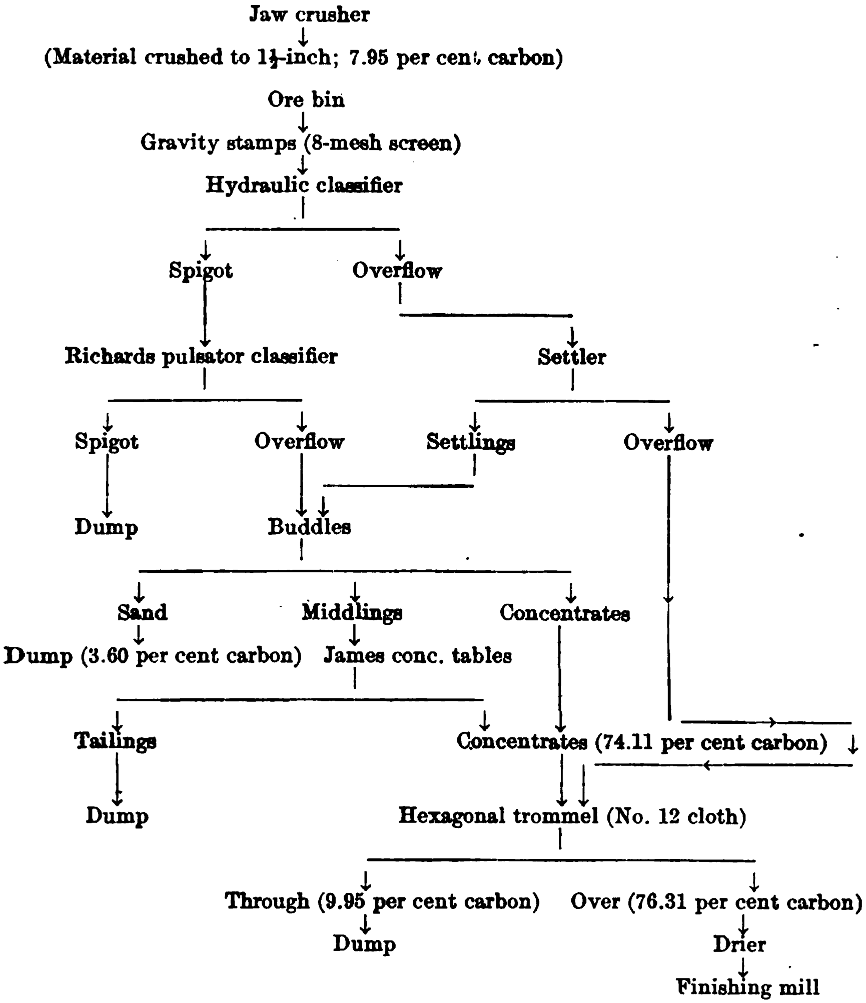
Jaw crusher
(Material crushed to 1¼-inch; 7.95 per cent carbon)
Ore bin
Gravity stamps (8-mesh screen)
Hydraulic classifier
Spigot
Overflow
Richards pulsator classifier
Settler
Spigot
Overflow
Settlings
Overflow
Dump
Buddles
Sand
Middlings
Concentrates
Dump (3.60 per cent carbon)
James conc. tables
Tailings
Concentrates (74.11 per cent carbon)
Dump
Hexagonal trommel (No. 12 cloth)
Through (9.95 per cent carbon)
Over (76.31 per cent carbon)
Dump
Drier
Finishing mill

FIGURE 12.—Flow sheet of New York concentrating plant.

With crude ore that analyzed 7.95 per cent graphitic carbon, "buddle" tailings analyzed 3.60 per cent C. Tailings from various other machines analyzed 3.96 to 9.95 per cent C., the latter product consisting of the fines through the rough concentrate trommel. The crude concentrate analyzed 76.31 per cent graphitic carbon.

It should here be noted that the "buddle" is being replaced at the Jos. Dixon Crucible Co. plant by a system of oil flotation. As the product from this mine goes into the manufacture of materials other than crucibles, it is desirable to get as high a recovery of graphite of various grades as possible per ton of ore.

REFINING METHODS.

Refining methods do not differ greatly from methods used in Alabama plants. One variation is the use of the Hooper air jig instead of air classification. The chief disadvantage ascribed to this jig is its small capacity.

The American Graphite Co. refinery at Ticonderoga prepares foreign and domestic graphite, both amorphous and crystalline, for use in the manufacture of commercial articles, such as paints, foundry facings, pencils, lubricating flake, and boiler compounds. The by-products obtained from this mill are all utilized in the manufacture of the commodities mentioned.

Data as to recoveries of No. 1 flake, No. 2 flake, and dust in New York practice are not available.

THE INDUSTRY IN PENNSYLVANIA.

PLANTS.

The graphite industry in Pennsylvania has in the past suffered from " wild-cat " and " get-rich-quick " schemes that for a time seriously interfered with the production in the Pickering Valley, Chester county. During the war, this district, in common with other producing districts, responded to the increased demand for graphite. In 1918, five plants were in operation, two close to Byers and three near Chester Springs. The most recent installation is the Huff electrostatic system at the plant of the Rock Crucible Graphite Co., the contemplated flow sheet of which is given in figure 13.

At present (August, 1919) graphite production in Pennsylvania is practically at a standstill.

OCCURRENCE OF ORE.

The ore, with one exception, is a soft disintegrated schist dipping from 35° to 50° and analyzing 3½ to 4½ per cent graphitic carbon. In some places most of the gangue has been removed by weathering, the graphite remaining behind as large coarse flakes.

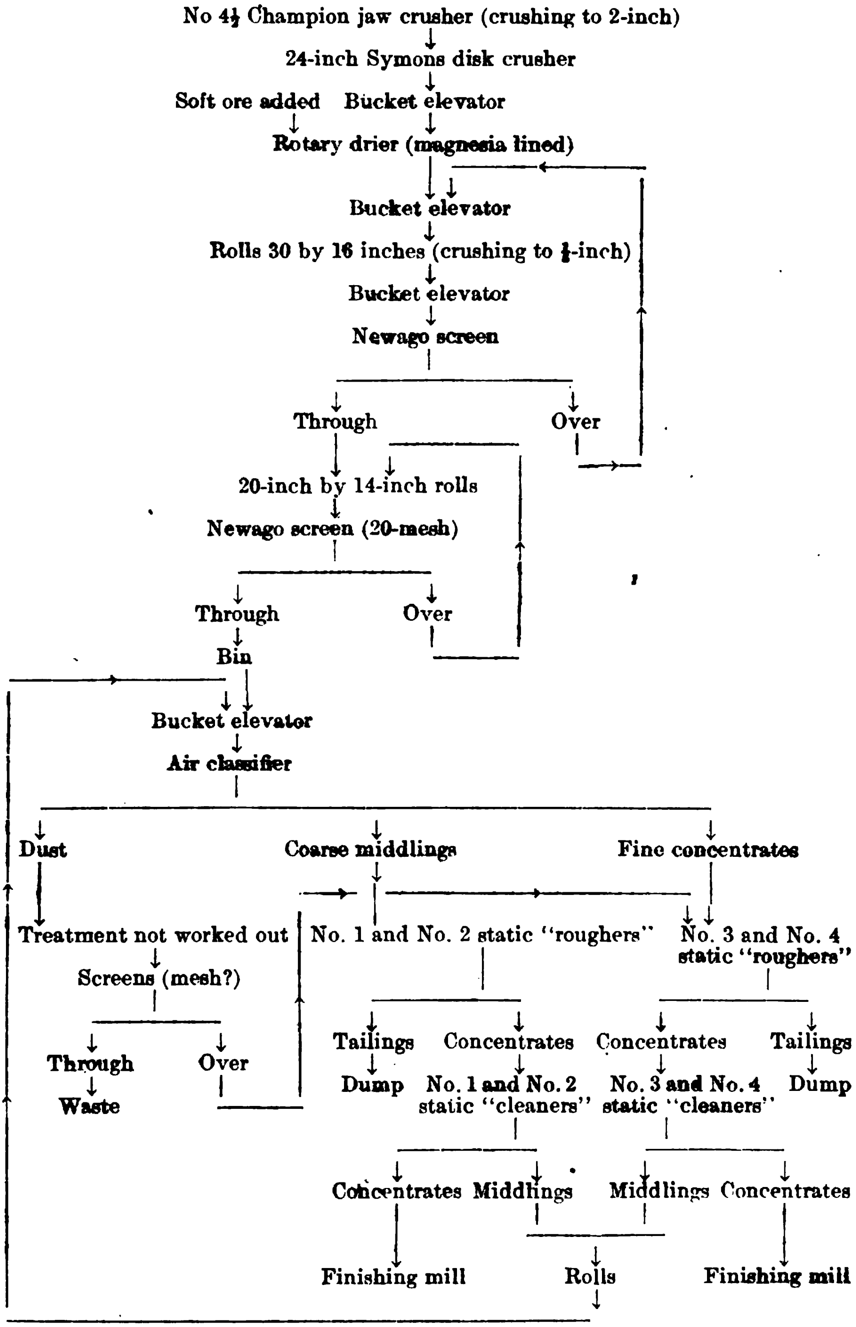

FIGURE 13.—Contemplated flow sheet for Huff electrostatic system.

MINING METHODS.

With the exception of the operations of the Standard Carbon Co. and part of the operations of the Rock Crucible Graphite Co. all mining is done in open pits. Loading of broken ore is done either by hand or by steam shovel, and little or no drilling is necessary because of the character of the ore.

At the mine of the Standard Carbon Co. the ore is mined in narrow rooms with alternating pillars. The deposit averages 25 feet thick and dips about 40°. The mine is developed by a 120-foot vertical shaft, with levels at 80 feet and 120 feet below the collar. The ore is readily drilled with an auger drill.

CONCENTRATING METHODS.

In this district three of the concentrating systems described under the outline of the Alabama industry are used. Three plants use the log washer or a similar device; one plant is using the dry process in the form of the Huff electrostatic system, and a fifth plant was using the oil-froth flotation system with the K and K type of cell, but has now discarded this type of cell.

At two of the plants a machine called the "oscillator" is used in place of the log washer. The "oscillator" comprises an inclined table with a series of rakes suspended over it and attached to an eccentric. In addition to the horizontal motion each row of teeth is given a circular motion in a vertical plane, thus stirring the pulp and releasing the graphite, which floats to the lower end of the table while the tailings are worked to the upper end by the rakes. In principle, therefore, this device resembles the log washer. As with the log washer, kerosene oil is used to help float the graphite. Samples taken in plants in which machines of this kind are used gave the following results:

Results of operation of oscillators.

[Figures represent percentages of graphitic carbon.]

	Test 1.	Test 2.
Crude ore	4.52	3.56
Washer tailings	2.19	1.79
Rough concentrates	62.00	72.07

The contemplated flow sheet of the plant in which the Huff electrostatic system has been installed is shown in figure 13. The other four plants have flow sheets similar to those described under Alabama practice.

REFINING METHODS.

Methods of refining used in the Pennsylvania field are similar to those in use in other districts. Two refining plants are being operated. In the new Huff electrostatic plant it is expected that no additional refining apparatus except dusting and bolting screens will be required. The plants not operating refineries sell their rough concentrate, representing material coarser than 100 mesh and analyzing 60 to 70 per cent graphitic carbon, to an Eastern refiner.

In this district data as to the recovery of finished products are not available. From 50 to 60 pounds of rough concentrate, analyzing as shown in preceding table, are recovered per ton of ore.

THE INDUSTRY IN TEXAS.

OCCURRENCE OF ORE.

In Texas graphite is found in the siliceous pre-Cambrian Packsaddle schist in the central region of the State between Llano and Burnet. In many places, the graphitic schist has been disrupted by granite and pegmatite intrusions. This schist, according to Paige,[a] is a metamorphosed sedimentary rock in which the carbonaceous materials have been recrystallized to form graphite.

The ore analyzes 6 to 10 per cent graphitic carbon, but the recovery of No. 1 flake is comparatively low because of the presence of amorphous graphite and very small flake in the ore; moreover, the gangue is hard, so that in crushing, a large amount of the flake is destroyed by being ground to finer sizes than are used as No. 1 flake.

In 1918, three properties were in operation in the State, but in June, 1919, only one was active.

MINING METHODS.

At the properties of the Southwestern Graphite Co., 9 miles west of Burnet and the Burnet-Texas Graphite Co., 7 miles west of Burnet, open pits have been developed; but at that of the Dixie Graphite Co., 4 miles northeast of Llano, mining is by underground methods.

At the property of the Southwestern Graphite Co., the overburden, which varies in thickness from 2 to 8 feet, is a hardbaked detritus containing large boulders of ore. It contains much clay and vegetable fiber and is said to average 3 or 4 per cent graphitic carbon, only a small part of which can be recovered as No. 1 flake stock. Accordingly, this material is loosened by means of a plow aided by a snatch block and is dumped as waste. In loosening overburden of this character, blasting powder might advantageously be used.

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[a] Paige, Sidney, U. S. Geol. Survey, Geol. Atlas, Llano-Burnet folio (No. 183), 1912, p. 4.

When the mine was visited the ore body was being crosscut, preliminary to the development of two working faces along the strike of the ore, which is 80 to 100 feet thick and dips about 65° southeast. The outcrop can be followed on this company's property with a slight interruption for almost three-fourths of a mile along the strike.

In mining, a Keystone wood-fired churn drill is used for drilling holes 5½ inches in diameter. These are placed at the corners of 12-foot squares the first row of which is 12 feet from the last crack of the previous blast. All holes are drilled to a depth of 25 or 30 feet, which is 5 feet below the elevation of the bottom of the open pit. Blasting gelatin of 60 per cent strength is used for breaking. An Erie wood-fired steam shovel with a dipper capacity of five-eighths of a cubic yard loads the broken ore into 5-ton side-dump contractors' cars which are hauled to the mill by a Whitcomb gasoline locomotive. Large boulders are placed to one side by the shovel and are block-holed with self-rotating hammer drills of the jackhammer type.

The Burnet-Texas Co.'s property is 2 miles northeast of that of the Southwestern Graphite Co. on the same schist series and at the time of visit was being worked in a small way. Here the ore is more frequently cut by granite and pegmatite intrusions, and as developed appeared to contain less graphite that would make No. 1 flake stock. Besides an open pit, this property was being prospected by a vertical shaft.

At the Dixie Graphite Co. property, 4 miles northeast of Llano, the ore is won by underground mining. The deposit dips to the southeast at a flatter angle than at the Southwestern Graphite Co.'s property. Near the surface the ore contains considerable quantities of amorphous graphite and fine flake. That mined underground is of better grade as regards the size of flake, and contains considerably more pyrite and mica than were obtained in the surface ore. In the past, much development work was done on this property with a view to mining gold and molybdenum ores, but these efforts were not successful.

CONCENTRATING METHODS.

Two plants use the oil-froth flotation method; at a third plant Huff electrostatic machines have been installed. These plants do not differ materially from mills previously described, except that in each a rough concentrate analyzing 70 to 80 per cent graphitic carbon and remaining on an 80 or a 90 mesh screen is recovered.

No refining plants are as yet in operation, although one company is reported to have completed alterations in its mill that will permit production of refined graphite.

In this district the same lack of storage facilities for crude ore was noted as in the Alabama field. The general remarks as to storage also apply to the New York and Pennsylvania districts.

RECOVERY OF GRAPHITE FROM "KISH."

The product known as "kish" is a mixture of graphite, slag, iron oxide, fragments of iron, and other materials that accumulate about a pig-iron casting ladle, or a Bessemer or open-hearth furnace. The graphite is formed during cooling by the crystallization of excess carbon from molten pig iron. The kish obtained for refining has contained 8 to 10 per cent of graphitic carbon in the form of a light, thin, and fluffy flake.

The method of concentrating and refining kish is similar in many ways to the methods employed with natural graphite. The chief points of difference are that kish is not subjected to crushing, but is carefully screened and is treated with magnetic separators. A patent (No. 1239992) on the recovery of graphite from kish has been issued to F. W. Weisman, and another patent (No. 1271146), to E. C. Ewen.

Kish as a possible source of graphite should not be overlooked. It is questionable, however, whether enough of this material can be obtained to interfere or compete seriously with the mining of natural flake graphite.

EXPERIMENTAL WORK ON CONCENTRATION AND REFINING.

As is evident from the foregoing discussion, much work had been done on various methods of concentrating graphite ore and refining graphite, but no large amount of effort had been expended on making comparative tests of the processes. It is evident, too, that the possibilities of recovering larger quantities of No. 1 flake have not been exhausted. For these reasons the Bureau of Mines has felt justified in conducting experiments at its Salt Lake City experiment station on the concentration of graphite ore and the refining of graphite. This work is described in the report of F. G. Moses, "Refining Alabama flake graphite for crucible use" (pp. 46 to 74).

EXPERIMENTAL WORK ON CRUCIBLE MANUFACTURE.

The Bureau of Standards at various times has done some work on crucible manufacture with a view to the use of materials other than the imported clays and graphites. This work has never been carried to a conclusion. It was felt therefore that the Bureau of Mines at its Columbus experiment station should make an investigation that would decide to what extent flake graphite might be used in crucible manufacture, and would establish physical and chemical specifications for domestic flake graphite.

PHOTOMICROGRAPHY.

As microscopic examination of crucibles made by various manufacturers, in conjunction with a knowledge of the crucible ingredients, may be of assistance in the solution of the mechanical difficulties attending the use of more than 25 per cent domestic flake graphite in crucible graphite mixtures, this work has been assigned to the Pittsburgh experiment station of the Bureau of Mines.

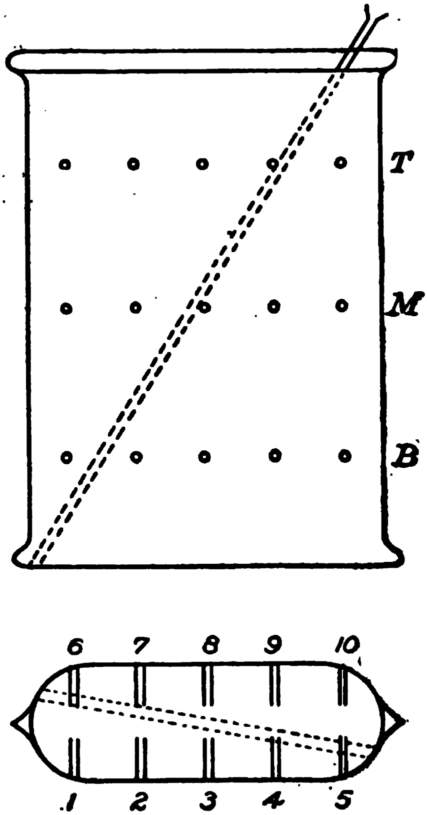

Figure 14.—Method recommended for sampling finished sacked product. Correct position of sampling device indicated by dotted lines in two views. The method now generally employed gives samples corresponding to 1 (T, M, B), 3 (T, M, B), or 5 (T, M, B).

METHODS OF SAMPLING.

Generally speaking, the only samples taken in graphite plants are of the finished products. In the past these have not been sampled in the same way by producer and by consumer, so that serious disputes over shipments have arisen, with the result that consumers have often refused to accept certain shipments.

By daily sampling crude ore and crude concentrates, as well as finished products, a much better check on mill work can be obtained than is possible by sampling only the finished grades.

Domestic graphite is usually marketed in sacks holding 150 pounds. The sacks are filled from a spout at the bottom of a small bin to which the products come from the final sizing screen, which follows the buhr mill. In the process of refining, a product absolutely homogeneous in carbon content is not being made at all times of the day. The material that goes to the finished-product loading bin often varies 5 per cent from the standard determined by the mill operator. The causes of these variations are many. Thus, products obtained just after the buhrstones have been freshly dressed are different from those obtained before the stones have been dressed;

and the feed to the buhrstones varies in character from time to time because of frequent concentrator shutdowns. The stones are adjusted from time to time during the day, but sometimes the character of the No. 1 flake obtained is visibly below the predetermined plant standard. As the sack is filled from a vertical spout, any fluctations in the grade of graphite in the bin appear as strata in the bag. Moreover the larger flakes naturally fall to the edges of the pile under the spout. The miller usually samples a bag of graphite by taking a spoonful of graphite from the top of the sack or by taking a sample at three points in the bag by means of a small hollow bag sampler about 8 inches long, with an opening 2 inches long near the point.

To show that these methods could not give a representative sample, two tests of graphite in sacks were made as shown in the table following and illustrated in figure 14.

Results of two tests of sampling sacked graphite.

[Figures and letters are same as in fig. 14.]

Position of sample.	Carbon.	
	Volatile.	Graphitic.
	Per cent.	*Per cent.*
T (1-5)	2.46	86.08
M (1-5)	1.78	87.32
B (1-5)	1.68	87.65
T (6-10)	2.25	84.74
M (6-10)	2.49	88.82
B (6-10)	2.10	87.63
1 (T, M, B)	2.56	88.93
3 (T, M, B)	2.26	84.90
5 (T, M, B)	2.67	83.71

In view of the considerations mentioned and the sampling results presented in the table, it is recommended that every bag be sampled continuously from top to bottom in the manner indicated in figure 14, by means of one of the devices described below.

One device consists of a tubular sleeve (fig. 15) 30 inches long and ⅜ to ½ inch in diameter, with a continuous slot extending from the point to within 4 inches of the top. Inserted in the sleeve is a rotating tube with a slot to correspond to the opening in the outer tube. Another suggested sampling device is a single tube with a check valve at the bottom for retaining the sample while the tube is being removed from the sack. It is further recommended that all samples be taken obliquely from top to bottom to get a fair proportion of the larger flakes which naturally slide farthest during the filling of the sack, and tend to collect at the sides.

The method of sampling shipments adopted by the Graphite Producers Association of Ashland, Ala., is an honest effort on the part of Alabama operators to avoid disagreeable and costly disputes with the consumer.

As soon as a quantity of graphite is ready for shipment, the local chemist at Ashland divides the shipment into lots of 25 sacks. Samples are drawn from each bag and then combined to represent the individual lots. Analyses are made of the lot samples, and if any lot falls seriously below shipping grade, it is discarded from the shipment. The grade of the shipment is then calculated by averaging the analyses of the lot samples.

METHODS OF ANALYSIS.

The method of analyzing graphite products or ores has never been standardized to the extent of defining volatile and fixed carbon in a manner acceptable to all analysts. Accurate methods used in determining carbon in graphite products are tedious. Work recently done by the Pittsburgh experiment station of the Bureau of Mines on the chemical analysis of graphite products had in view the devising of a rapid, accurate, and simple method of analysis with a fair degree of accuracy. A description of this method is given on pages 43 to 45.

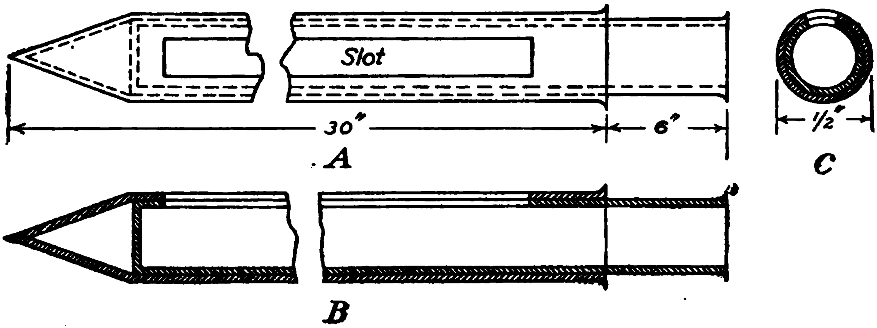

FIGURE 15.—Sections of sampling device (not drawn to scale). *A*, Longitudinal section, showing slot; *B*, longitudinal section, samples turned 90° from position A; *C*, cross section through slot.

TENTATIVE SPECIFICATIONS.

In the past, crucible makers who used domestic flake graphite have as a rule bought this material only after examining samples submitted by producers. It may be helpful to suggest tentative specifications for No. 1 flake graphite.

The table following gives the screen and chemical analyses of graphite that is being used in the experimental work of the Columbus experiment station of the Bureau of Mines on graphite crucibles. All screen analyses were made at the Pittsburgh station of the bureau. A Tyler standard Ro-tap apparatus was used, and the graphite sample was screened for 45 minutes. All chemical analyses were made at the Pittsburgh station, the volatile-carbon determinations of samples being made by heating for three minutes at 800° C.

Results of analyses of domestic flake graphite.

Sample No.	Kind of sample.	Carbon.		Cumulative percentages.				
		Volatile.	Graphitic.	Over 20 mesh.	Over 35 mesh.	Over 65 mesh.	Over 100 mesh.	Under 100 mesh.
		Per cent.	*Per cent.*					
1	Artificial graphite	1.27	87.82	0.3	11.7	54.9	95.9	4.1
2	No. 1 flake	1.68	82.02	....	.1	42.0	94.8	5.2
3	....do	1.32	92.09	....	2.5	28.0	60.0	40.0
4	No. 1 flake and No. 2 flake	2.07	86.85	....	3.0	31.0	74.3	25.7
5	No. 1 flake	3.65	74.67	....	8.3	80.3	89.6	10.4
6	....do	....	....	.1	15.4	69.6	95.6	4.4
7	Dust	6.06	41.33	....	.1	1.2	3.2	96.8
8	....do	7.37	32.41	....	....	....	.2	99.8
9	....do	7.20	31.47	....	....	.4	3.9	96.1
10	No. 2 flake	2.96	80.44	....	....	....	10.5	89.5
11	....do	2.31	81.43	....	.2	9.9	67.6	32.4
12	No. 1 flake	....	....	....	5.7	58.5	93.0	7.0
13	....do	1.98	86.18	.1	6.7	56.4	97.0	3.0
14	No. 2 flake	2.17	86.30	....	.1	.3	22.2	77.8
15	No. 1 flake	....	....	....	9.7	62.2	92.4	7.6
16	No. 1 flake, special	1.08	92.43	....	8.2	69.6	99.7	.3

Nine crucible manufacturers sent to the Bureau of Mines samples of graphite as prepared by them for incorporation in their crucible mixtures. Screen analyses of these samples were made at the Washington laboratory; the results showed considerable variation. The laboratory lacked the equipment usually found in establishments for the making of screen analyses, and consequently a nest of sieves of 20, 40, 60, 80, and 100 mesh had to be used rather than the standard meshing. For comparison, however, the results will answer just as well.

Results of analyses of manufacturer's samples at Washington laboratory.

No. of sample.	Manufacturer.	Cumulative percentages.						Volume of 100 grams (c. .c).	Remarks.
		20 mesh.	40 mesh.	60 mesh.	80 mesh.	100 mesh.	100 mesh.		
1	A	1	68¼	83½	92¼	94¾	100¼	106	Contains 35 per cent flake and 65 per cent Ceylon.
2	B	13½	57¾	71½	80¾	85½	99½	76	100 per cent Ceylon.
3	C	12¾	88½	96½	96½	99	100	149	Madagascar flake.
4	C	16½	71	81½	89½	90¼	100	86	100 per cent Ceylon.
5	D	13¾	62½	74¾	88	93¾	99½	81	Contains 10 per cent flake and 90 per cent Ceylon.
6	E	23¼	66	76	83½	87	100	81	100 per cent Ceylon for steel "pot."
7	F	1¼	37½	62¼	88¾	96½	100	145	100 per cent Canadian.
8	G	14¼	58¼	75	85¾	89¼	99½	95	Contains 25 per cent flake; 75 per cent Ceylon for steel "pot."
9	H	4¼	49¼	69	80½	84¾	99¼	93	Contains 15 per cent flake and 85 per cent Ceylon.
10	I	40	74	84	89½	92	99½	89	100 per cent Ceylon.
11	I	16½	59½	75¾	86¾	90	99¾	116	Contains 17 per cent flake and 83 per cent Ceylon.

CARBON CONTENT.

It is recommended that the graphitic carbon content of No. 1 flake be not less than 85 per cent. By graphitic carbon is tentatively meant the carbon remaining after the dried sample has been burned for three minutes at 800° C.

The table following gives the results of complete analysis of seven samples of crucible graphite. The analyses were made at the Pittsburgh station of the Bureau of Mines. The volatile carbon was determined by heating the samples at 800° C. for three minutes.

Results of complete analysis of seven samples of graphite.

Source of graphite	Alabama.	Alabama.	Alabama.	Alabama.	New York.	Pennsylvania.	Ceylon.
Sample No.	1	2	3	4	5	6	7
Volatile carbon	2.08	1.40	2.66	1.31	1.30	1.53	1.68
Graphitic carbon	84.52	90.58	81.32	91.18	88.97	88.80	85.06
SiO_2	7.02	3.00	8.83	4.08	4.34	5.24	7.81
Al_2O_3	5.06	2.96	6.35	2.31	2.40	2.05	2.82
Fe_2O_3	.53	.18	.39	.40	1.08	1.75	1.61
TiO_2	.14	.19	.13	.13	.38	.05	.13
CaO	....	....	....	....	.07	....	.19
MgO	.23	.16	.06	.08	.76	.09	.21
K_2O	.15	.23	.14	.31	.55	.08	.25
Na_2O	.008	.01	.11	.03	.12	.12	.11
SO_3	.007	.14	....	.01	....	.21	.005
P_2O_5	.06	....	....	.02	.02	.05	.05
MnO	....	....	....	....	....	.07	.04
CuO[a]	....	.14	....	....	....	....	....
ZnO[a]	....	....	....	....	....	....	.03
Total	99.805	99.98	99.99	99.86	99.99	100.04	99.995

[a] Contamination probably from brass screens.

The results presented indicate that the amount of deleterious impurities in domestic flake graphite is small. In the samples of Alabama graphite the Fe_2O_3 content did not exceed 0.53 per cent, and the combined content of alkalies and alkaline earths did not exceed 0.5 per cent. The amounts of mica and pyrite present were small indeed, judged from the alkali and SO_3 content. Even the Pennsylvania sample with a high Fe_2O_3 content showed only a small amount of SO_3, also a low alkali content. The sample from New York showed a combined alkali content of 1.5 per cent, and the Fe_2O_3 content was slightly more than 1 per cent.

If the single sample of Ceylon graphite analyzed may be taken as representative—and it should be stated that this sample was graphite prepared for use in crucibles—it is evident that, chemically, domestic graphite compares favorably with the Ceylon graphite.

SCREEN ANALYSIS.

The following tentative specifications as to screen analysis in cumulative percentages are recommended:

Recommended specifications for screen analysis.

[Figures represent cumulative percentages.]

Grade	No. 1 flake.
Over 35 standard mesh	35
Over 65 standard mesh	50
Over 100 standard mesh	100

Permissible allowance of not more than 3 per cent through 100 mesh.

PRESENT STATUS OF GRAPHITE MINING.

The request of the War Industries Board on August 10, 1918, that not less than 20 per cent of domestic flake graphite be incorporated into crucible-graphite mixtures during the balance of 1918, and 25 per cent thereafter, established a market for domestic flake which, it was hoped, would continue after the war. If domestic graphite can be used in this proportion without adversely affecting the quality of crucibles, it will be used provided it can be produced at costs that compare favorably with the prices paid for imported graphites. Under normal conditions the efficiency of crucibles is expressed as so many heats per crucible. However, during the war the vital comparison, as regards the national welfare, was the number of pounds of imported graphite used per heat. The introduction of every pound of domestic flake graphite that did not increase the consumption of imported graphite per crucible heat saved necessary ship tonnage. The proportion of domestic flake that reduces the consumption of imported graphite to a minimum has not yet been determined.

In all of the graphite mining districts in this country operating costs are very high, and in normal times, if imports were unrestricted, the domestic mining industry could not compete with the industries in Madagascar and Ceylon. On both of these islands, the deposits are of large extent and of such high grade that a simple cobbing or washing represents the only milling necessary. Labor is much cheaper in the Far East than in the United States. Therefore, in spite of the great distance from the market, graphite from these islands could be placed on the docks in this country at prices which at present would provide no profit for domestic producers. Also, overproduction abroad might tend to force prices still lower through dumping of surplus production on the American market.

Operating costs in the United States in October, 1918, ranged from 6 to 14 cents, with an average of 10 cents, per pound of No. 1 flake. No allowance has been made for depletion and depreciation, which would add 1 to 2 cents per pound to the costs stated. In arriving at these costs the production of No. 1 flake has been charged with all operating expenses and has been credited with the miscellaneous income derived from the sale of No. 2 flake and dust. The wide variation in costs is due to differences in the efficiency of individual plants as well as to fluctuations in the grade of ore treated by the different plants.

The graphite in the Alabama district is of lower grade than that in any of the other districts mentioned in this report. With an average recovery of 19 pounds of No. 1 flake per ton of ore and an after-war price of 9 cents per pound, which is 3 to 4 cents higher than the pre-war price, it is evident that, even though all the ore is mined in open pits, only those plants efficiently managed and working comparatively rich deposits will survive unless new and important uses develop for the by-product grades so as to warrant the establishment of a local central factory for producing commercial graphite products. Many of the Alabama plants are 6 to 9 miles from the railroad and are in localities where most of the roads are difficult to maintain because they are of clay and poorly drained. At plants so situated the cost of transportation becomes a serious factor in operating expenses.

Until 1913, the graphite production in Alabama was small, but has steadily increased so that since 1915 Alabama has been the leading graphite-producing State in the Union.

Milling capacity in the Alabama district, with the 39 plants now in operation or building, is sufficient to produce a large quantity of graphite, if these mills could all run 20 hours a day. On the assumption that each mill would average 3,000 pounds of No. 1 flake daily, which is a conservative estimate, a total production of nearly 60 tons a day could be obtained. The actual production of No. 1 flake from this district during 1918 averaged less than 350 tons per month.

In New York and intermittently in Pennsylvania, graphite was mined before the war. New York will undoubtedly continue to produce graphite after the war in somewhat greater quantities than before. Both of these districts are close to a large market. The recovery of graphite per ton of ore is double that obtained in Alabama; this is a distinct advantage, particularly when by-product grades can be sold advantageously.

The costs of production are practically the same as those in Alabama, chiefly because most of the plants in New York and Pennsylvania have just started producing; but with reversion to peace times, these districts have a distinct advantage over the Southeastern district, in spite of the necessity in New York for underground mining.

In the latter part of 1918 the New York district was producing not more than 90 tons of graphite per month, of which probably 60 per cent would be No. 1 flake. The Pennsylvania field produced about 50 tons of graphite per month, of which about 60 per cent would be No. 1 flake.

The Texas district, if provided with an outlet for by-product grades of flake graphite, would have a good opportunity of surviving post-war readjustments. The production from this district has not been large, but could be considerably increased without great difficulty. With respect to a Western and Central market, the Texas deposits are favorably situated. The deposits are not only of large extent but are high grade as well.

These statements have been made in order to indicate, in a general way, the precarious position of the mining of domestic crystalline flake graphite. It would be presumptuous to suggest methods by means of which the mining of domestic graphite can be established firmly on a postwar basis, for underlying the entire consideration is the fundamental economic law of supply and demand. As regards domestic flake graphite, the supply exists and can be obtained. If the demand can be created or extended by scientific, rather than by artificial means, the supply will be forthcoming whenever the price is high enough to justify production.

With the resumption of peacetime activities, the demand for crucibles and other graphite products has slackened. This, of course, has reacted upon the domestic graphite mining industry, with the result that production has been greatly curtailed. Until business in the United States has resumed its normal peace-time trend, some method of control or regulation will be necessary to prevent the collapse of certain industries, which the lessons of the present war have taught should not be permitted to die. To be independent of foreign graphite, the use of 100 per cent domestic flake in crucibles will have to be developed, if the graphite crucible continues to be used as a medium for melting alloys of all kinds. It is possible, of course, that the use of some refractory other than graphite, or the extended use of melting furnaces requiring no crucibles may develop to such a point as to make present practice obsolete.

METHOD FOR RAPID ANALYSIS OF GRAPHITE USED BY THE BUREAU OF MINES.

By G. B. Taylor and W. A. Selvig.

In the following paragraphs are outlined certain methods of graphite analysis used by the Pittsburgh laboratory of the bureau. These are intended to provide a simple and rapid procedure for the proximate analysis of graphite, such as can be performed in the average laboratory in the minimum of time, together with a fair degree of accuracy. The proximate analysis consists of the determination of moisture, volatile matter, ash, and graphitic carbon by difference.

PREPARATION OF SAMPLE.

On account of the difficulty of grinding flake graphite, no degree of fineness can be fixed; however, for material containing considerable gangue, grinding to pass through a 60-mesh sieve is recommended. The flakes of graphite separated during the screening must of course be included in the 60-mesh material, and must be thoroughly mixed before portions for analysis are weighed out.

DETERMINATION.

MOISTURE.

One gram of the sample is placed in a weighed porcelain capsule ⅞-inch deep by 1¾ inches wide, and heated for one hour at 105° C. in a constant-temperature oven. The capsule is then removed from the oven, covered, and cooled in a desiccator over sulphuric acid. The loss in weight multiplied by 100 is recorded as the percentage of moisture. If platinum crucibles are available, it is preferable to use a 25-c.c. platinum crucible instead of the porcelain capsule.

VOLATILE MATTER.

The covered capsule containing the graphite from the moisture determination is placed in a muffle furnace and heated at 800° C. for three minutes, removed from the furnace, cooled in a desiccator, and weighed. The loss in weight multiplied by 100 is recorded as the percentage of volatile matter.

A lower temperature and a shorter time interval than is used in the standard method for the determination of volatile matter in coal is recommended, as the volatile-matter content in graphite is small, and the oxidation and loss of graphitic carbon should be cut down to a minimum.

ASH.

Ash is determined in the residue from the volatile-matter determination. The capsule containing the residue from that determination is placed in a muffle furnace at about 800° C. and is heated until completely burned. The capsule with its contents is removed from the muffle and cooled and weighed, after which it is replaced in the muffle, heated for half an hour, cooled in a desiccator, and weighed again. This heating and weighing should be continued until the change in weights between two consecutive ignitions is 0.0005 gram or less.

The weight of the crucible plus the ash, minus the weight of the empty crucible, multiplied by 100 is recorded as the percentage of ash.

GRAPHITIC CARBON.

Graphitic carbon, which is determined by calculation, is the difference obtained by subtracting the sum of the percentages of moisture, ash, and volatile matter from 100.

GRAPHITIC CARBON BY COMBUSTION.

The most accurate method of determining graphitic carbon is by combustion, and subsequent weighing of the CO_2 formed. While it has been found that graphitic carbon, as calculated, checks very closely with the graphitic carbon determined by direct combustion, the latter should be used when greater accuracy is desired. This method, however, calls for special equipment and requires considerable skill in manipulation.

On account of the possible presence of carbonates in ore, and possibly small amounts of flotation oils in the sample, if a concentrate, a preliminary treatment before combustion is required.

PRELIMINARY TREATMENT OF ORES.

A sample, weighing 0.2000 to 1.0000 gram, depending on the relative amount of graphitic carbon, is placed in a 100-c.c. evaporating dish with 25 c.c. of 1 to 1 HCl, heated over a hot plate for 15 minutes, filtered through a filter of ignited asbestos, and washed with hot water until free from chlorides. The filter and the residue are then transferred to a porcelain or platinum boat and dried on the hot plate. The boat containing the residue is transferred to the tube of a combustion furnace and burned in a stream of oxygen gas, the CO_2 formed being collected in a potash bulb with a 30 per cent KOH solution. The combustion tube should contain some fused lead chromate to retain any sulphur that may be present.

PRELIMINARY TREATMENT OF CONCENTRATES.

Concentrates are likely to contain flotation oils, which must, of course, be removed before combustion, or too high results will be obtained for graphitic carbon.

This may be done by placing a sample weighing 0.2000 to 0.5000 gram in a small Erlenmeyer flask and adding about 25 c. c. of ether, corking loosely, and allowing to stand for about one-half hour, shaking at intervals. The mixture is filtered onto asbestos in a Gootch crucible, the asbestos having previously been washed with hydrochloric acid and ignited. The residue on the filter is washed with alcohol and next with distilled water; it is then ready to be treated with acid for the removal of possible carbonates, as is done in the treatment of ores.

REFINING ALABAMA FLAKE GRAPHITE FOR CRUCIBLE USE.

By Frederick G. Moses.

INTRODUCTION.

In order to determine the possibility of producing a satisfactory crucible graphite from Alabama flake graphite, an investigation embracing a large number of tests was made at the Salt Lake City station of the Bureau of Mines. The following report contains the information obtained during the progress of this investigation.

A primary object of the work was to ascertain the most satisfactory means by which the low-grade graphite concentrates produced from Alabama ores could be made into satisfactory crucible stock. Of course, the success of any one of the methods of treatment developed will depend both on the amount of satisfactory material that can be recovered by the finishing process, and on the ease and cheapness of the operation itself. In all of the experiments both of these factors were kept in mind.

REQUIREMENTS FOR CRUCIBLE GRAPHITE.

After an extended investigation into the specifications for graphite satisfactory for use in crucible manufacture, Geo. D. Dub, has suggested the following specifications for satisfactory No. 1, Crucible Flake[a]. For the sake of emphasis they are summarized here.

Dub suggested that a No. 1 flake is satisfactory, as regards carbon content when the content of graphitic carbon is not less than 85 per cent. Graphitic carbon is here intended to mean that carbon which remains after burning a dried sample three minutes at 800° C.

It is also suggested that 35 per cent of the product should remain on a 35-mesh standard screen, 30 per cent should pass through a 35-mesh and remain on a 65-mesh standard screen, and the remaining 35 per cent should be between 65 and 100 mesh. An allowance of 8 per cent through 100 mesh could be permitted.

The aim of the investigation herein reported was to produce material of the grade outlined from the concentrates of graphite from the Alabama graphite fields.

PRESENT PRACTICE.

The production of crucible stock from the Alabama ores involves two distinct steps. The first is a concentration process by which a comparatively low-grade concentrate is recovered from the ore. The second step comprises the conversion of this low-grade concentrate into a high-grade material that will be satisfactory for crucible making.

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[a] See page 40 of this bulletin for "carbon content" and page 39 for "screen analysis."

The first step, being simply concentration, need not be discussed here. The second step, finishing, as it is called, is much more difficult to carry out and is the one that demands the greatest amount of investigation and improvement.

The present method of finishing the crude concentrate consists of, drying the concentrate, grinding the dried material in a buhr mill, and "bolting" or screening the ground product to remove the impurities that have been crushed during the grinding. The theory is that the graphite particles, being tougher, smoother, and thinner than the impurities, will be rubbed and polished between the buhr-stones with a minimum reduction in size, while the impurities themselves, owing to their more brittle characteristics and more massive structure, will be ground fine enough for removal by screening over a suitable cloth.

The method is simple, and in fact, within certain narrow limits and under favorable circumstances, gives good results. As a general rule, however, two difficulties are encountered that interfere with its commercial success—the grinding often destroys a large proportion of the coarse and valuable flake and there is a lack of uniformity in the finished product. The primary object of the present investigation was to determine the most efficient manner of obtaining the desired results and eliminating the troubles in the method mentioned above.

CONCENTRATES USED IN TESTS.

As has been suggested, the problem involved is the treatment of the concentrates that are being produced in the Alabama district. Therefore, all of the work done during this investigation was on a series of representative samples from that district.

The concentrates produced in the Alabama district differ widely in their physical characteristics and graphitic content. However, as the physical properties of the different minerals composing the concentrates can not vary to any considerable extent, a scheme worked out for any one concentrate can be applied, with only slight variations and adjustments, to the other concentrates. For this reason, and because of the numerous schemes tried in the limited time available for the work, it was impossible to conduct tests of all of the concentrates. Therefore, it was decided to select several of the most representative for investigation, and to determine the extent that the results obtained on these concentrates could be generalized and applied to all.

Table 1 gives the results of screen analyses and assays of the different samples selected.

TABLE 1.—*Results of screen analyses and assays of different samples selected for experiment.*

Mesh.	Percentage by weight.	Carbon assay (per cent).	Units of carbon, each size.[a]	Percentage of total carbon.
Sample 1:				
14	0.1	....	....	....
20	1.1	74.8	82.0	1.82
28	5.4	60.8	328—	7.27
35	14.8	53.6	793.0	17.60
48	22.5	47.2	1,062.0	23.55
65	21.8	44.4	968.0	21.47
100	17.4	44.00	766.0	16.99
150	8.8	43.40	382.0	8.47
200	2.7	41.20	111.0	2.46
−200	4.5	3.60	16.0	.35
Total	....	....	4,508.0	99.98
Sample 2:				
14	1.8	6.1	11.0	.21
20	2.4	15.7	37.7	.74
28	5.1	38.3	195.4	3.81
35	10.2	51.9	529.0	10.32
48	20.6	56.7	1,169.5	22.81
65	22.6	58.2	1,313.0	25.61
100	24.7	55.2	1,361.5	26.55
150	8.8	46.7	407.0	7.94
200	1.4	46.8	65.7	1.28
−200	2.3	16.2	37.2	.73
Total	....	51.27	5,127.0	100.00
Sample 4:				
14	Tr.	....	....	....
20	Tr.	....	....	....
28	.5	24.0	12.0	.37
35	6.2	20.7	128.0	3.92
48	16.1	25.9	417.0	12.79
65	23.1	33.1	765.0	23.46
100	25.7	43.4	1,114.0	34.16
150	16.1	49.1	780.0	23.92
200	4.0	56.1	22.0	.67
−200	6.5	36.9	23.0	.71
Total	....	....	3,261.0	100.00
Sample 5:				
14	.4	....	....	....
20	1.8	....	....	....
28	5.5	52.8	290.4	4.26
35	14.3	69.6	995.3	14.60
48	25.7	72.4	1,860.7	27.29
65	26.3	73.4	1,930.3	28.32
100	21.0	67.4	1,415.4	20.76
150	4.5	64.8	291.5	4.29
200	.3	....	....	....
−200	.9	36.0	32.4	.48
Total	....	....	6,816.0	100.00
Sample 6:				
14	....	....	....	....
20	.4	....	....	....
28	2.1	60.2	126.42	2.98
35	7.7	53.6	412.72	39.82
48	16.4	48.2	790.48	18.27
65	22.4	45.2	1,012.48	23.82
100	24.5	35.6	872.20	20.55
150	17.0	38.4	652.80	15.42
200	4.0	39.0	156.00	3.68
−200	5.5	40.4	222.20	5.35
Total	....	....	4,245.30	99.89

[a] Column 2 × column 3.

The characteristics of the samples were briefly as follows:

No. 1. A fairly coarse concentrate assaying 48.95 per cent carbon.

No. 2. A coarse flake concentrate assaying 56.47 per cent carbon.

No. 4. A fine flake concentrate assaying 42.38 per cent carbon.

No. 5. A medium coarse flake concentrate assaying 73.30 per cent carbon.

No. 8. A medium coarse flake concentrate assaying 42.45 per cent carbon.

As the work progressed, it was found that duplication of all of the necessary experiments on even this number of samples would involve too much time; hence two of the most representative were selected for investigation, and the conclusions drawn from the results obtained from them were applied to the rest. The two samples chosen for the work were Nos. 2 and 4, as they represented two general classes of concentrate—a fine and a coarse medium-grade material.

PHYSICAL PROPERTIES OF GRAPHITE AND ASSOCIATED GANGUE MINERALS.

It is evident that production from the concentrates described of a graphite stock containing 85 per cent or more carbon would first involve separation of the graphite from its contained impurities. This can be accomplished only by taking advantage of differences between the physical properties of the graphite and of the associated impurities. For comparison of the physical properties that might be utilized for this purpose they have been tabulated in Table 2, as follows:

TABLE 2.—*Physical properties of graphite and of associated minerals.*

Mineral.	Specific gravity.	Hardness.	Brittleness.	Shape.	Color.	Electroconductivity.	Surface condition.	Cleavage.
Graphite.	2.1	1 to 2	Not brittle..	Flat, tabular plates.	Dark gray and black.	Good....	Metallic luster.	One direction.
Quartz...	2.66	7	Very brittle.	Massive, granular, or compact.	Colorless, or may be any color.	Nonconductor.	Glassy or vitreous.	Practically absent.
Mica.....	2.8	2 to 2½	Not brittle..	Thin, scaly plates.	Colorless, or pale green to brown to black.	...do.....	Smooth, glassy, or vitreous.	Nearly perfect in one direction.

Some of these properties vary greatly, but others are closely alike. Unfortunately the property of specific gravity, most often used in the separation of minerals, can not be used in finishing graphite because the weights of the various substances involved are nearly the same. On the other hand, properties that are the least often used in the commercial separation of minerals, for instance, electrical conductivity, are the properties that show the greatest amount of difference and must be depended upon to give the best results.

Thus the standard equipment manufactured for the separation of minerals, such as gravity tables, can not be used to the best advantage in finishing graphite. In fact, past experimental work with specific gravity machines has substantiated this conclusion. Also, because no satisfactory equipment has been developed to make a separation by taking advantage of the little used physical characteristics, it was necessary to construct special apparatus.

ASPIRATOR TESTS.

Each apparatus investigated during this work uses the physical properties or characteristics of the graphite and gangue minerals as a means to accomplish separation.

The first experiments were made to determine the possibility of separating the graphite and gangue material by differences in specific gravity and shape of the various particles. The apparatus first tried depends on the difference in specific gravity and the difference in shape of the various minerals to accomplish separation. This type of apparatus is already being used to a limited extent in Alabama and is there known as an "aspirator."

The aspirator is fundamentally an air classifier. This name is appropriate for the reason that the separation depends on the settling rate of the different minerals in a current of air.

The apparatus (Pl. I, *A*) constructed for the experiments was closely similar to those that are being used in the Alabama field at the present time. The material to be treated is fed from a hopper to a roller from which it drops into the large end of a long, sloping, horizontally placed box. The roll feeder is arranged so as to feed the material in an even curtain across the whole opening. From the small end of the box a pipe leads to a suction fan. The box is 13.5 inches wide by 12.5 inches high at the feed end, and 43.5 inches long. The suction pipe is 31 inches long and 4 inches in diameter.

Across the opening of the box and parallel to the ground line are four vanes or sand deflectors, placed at such an angle from the vertical that any material fed from the roller when the air current is off will be deflected to the outside of the box.

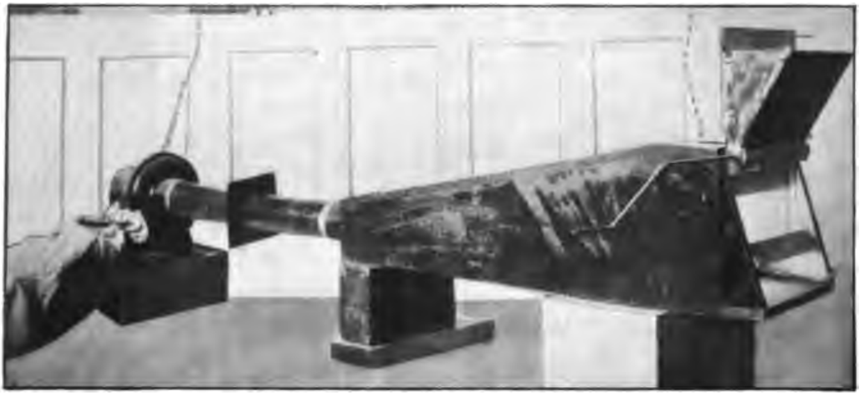
For a description of the operation of the classifier see page 25.

Several samples of the concentrates already described were tested with this machine, representative results of several runs being as follows:

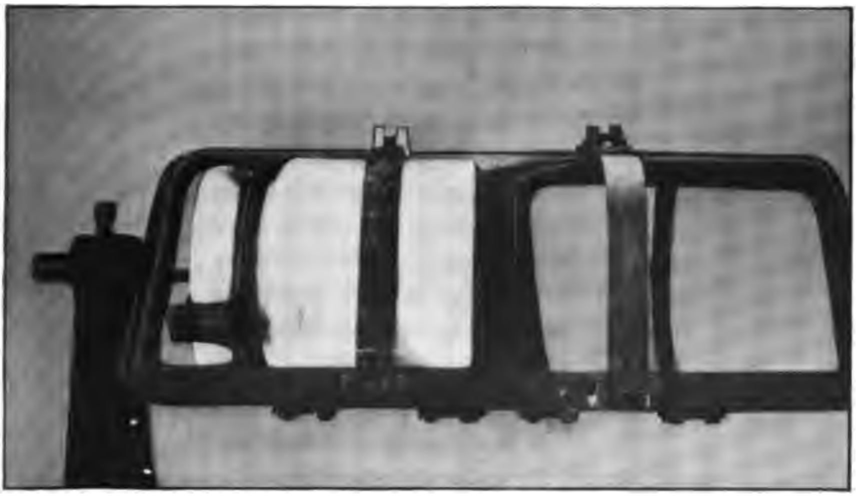
	Graphite, per cent.
Heads	51.23
Concentrates	69.23
Tailings	19.08

These results show a calculated recovery of 86.25 per cent of the total graphite in the concentrates produced.

The aspirator yields three products, as follows: Sand tailings, which are recovered on the outside of the box below the feed hopper; coarse graphite concentrates, which are heavy enough to be carried into the box and are deposited there, and the pure, fine concentrate



A. IMPROVISED ASPIRATOR USED IN TESTS AT SALT LAKE CITY STATION.



B. BUHR MILL USED IN GRAPHITE GRINDING TESTS.

that is too light to be deposited within the box, but is sucked through the blower and caught in a bag at its outlet.

In the particular test described, 87.1 per cent of the concentrate was deposited in the box and the remaining 12.9 per cent was drawn through the fan. The former material assayed 68.4 per cent graphite; the fine material assayed 74.85 per cent.

In order that the difference in the sizes of the various products made may be compared with the material before treatment, and with each other, the results of screen analysis and of assay of each of the sizes are given in Table 3 following. In this table the results for fine and coarse concentrates were combined.

TABLE 3.—*Results of screen analyses and of assays of aspirator concentrates.*

COARSE CONCENTRATES.

[87.1 per cent of aspirator concentrates.]

Mesh.	Proportion by weight.	Carbon assay.	Carbon, units.[a]	Total carbon.
	Per cent.	*Per cent.*		*Per cent.*
14	Tr.			
20	0.4	83.7	33.5	0.49
28	2.6	89.0	231.8	3.39
35	10.6	87.4	926.5	13.55
48	24.4	80.8	1,972.0	28.83
65	25.4	69.0	1,753.0	25.63
100	28.3	62.0	1,756.0	25.68
150	7.1	24.4	173.2	2.53
200	.5	23.3	11.5	.17
−200	.5	23.4	11.5	.17
Total	99.8	68.39	6,839.0	100.44

[a] Column 2 times column 3.

FINE CONCENTRATES.

[12.9 per cent of aspirator concentrates.]

14	None.			
20	None.			
28	Tr.			
35	1.6	88.1	141.00	1.70
48	9.2	90.3	831.00	10.92
65	24.1	87.8	2,118.00	28.11
100	37.8	81.1	3,063.00	40.74
150	17.1	62.8	1,073.00	14.17
200	3.9	48.8	190.00	2.36
−200	5.8	29.2	169.00	2.08
Total	99.5	74.85	7,585.00	100.08

ASPIRATOR TAILINGS.

14	5.00	4.1	20.5	1.05
20	8.00	12.9	103.2	5.41
28	12.80	23.2	297.5	15.59
35	18.50	26.9	498.0	26.10
48	28.40	24.0	681.5	35.69
65	20.00	13.2	264.0	13.83
100	6.40	5.9	37.8	1.98
150	.20	12.3	2.5	.13
200	Tr.			
−200	.20	15.3	3.0	.15
Total	99.5	19.08	1,908.0	99.90

It should be noted that the greatest concentration and improvement of product is in the coarser material of the coarse concentrates. The great difference between the carbon content of the material coarser than 150 mesh and of that finer than this size is very striking and also important.

As regards the fine concentrates produced the large percentage (83.02), which is —48 mesh and +150 mesh, is noteworthy as is the high carbon content of the +200 material.

Treatment of another sample, No. 4, in the aspirator gave the following results, showing a calculated recovery of 96.2 per cent of the total graphite in the concentrates produced:

	Carbon, per cent.
Heads	42.61
Concentrates	56.62
Tailings	6.03

These results indicate that although some of the impurities can be removed with the aspirator, thereby materially raising the grade of the material, the application of this type of aspirator will never be general.

It has long been known that the coarse sand particles cause the greatest destruction of the large flake in the buhr mill. When the concentrates carry a large proportion of sand particles of such a nature that the type of treatment described will raise the grade of the crude product to 70 per cent or better, the aspirator can undoubtedly be used to great advantage before treating the concentrates in the buhr mill. However, unless there is a comparatively large proportion of such impurities in the crude concentrate, the aspirator can not be used to the best advantage. The apparatus should certainly not be incorporated in a finishing mill except after thorough and comprehensive tests to determine the results that can be obtained by its use, and then only on the advice of a competent engineer conversant with the conditions that must be met by the installation.

Probably the most potent reason for the comparatively poor results obtained with the aspirator was the wide variety in size of the material treated. When a current of air strong enough to remove the coarse graphite flakes from the feed was used, the fine sand would, at the same time, be drawn into the box with the concentrate.

However, when the concentrates are of low grade and contain a large quantity of coarse sand, treatment by such a simple process as the one just described is so advantageous that its use should be considered, and when feasible, the designing engineer should include an aspirator in a projected plant.

A. PNEUMATIC JIG USED IN TESTS.

B. BUHR MILL USED IN TESTS.

TESTS WITH PNEUMATIC JIG.

The pneumatic jig was the next apparatus tested. Its construction differs greatly from that of the aspirator, although the principles involved in the operation of each are similar. As this equipment has been manufactured on a commercial scale for several years, its use would be advantageous when feasible, as a more simple and standard "layout" could be arranged than where improvised equipment like that described was used. Both types would be simple to operate, the aspirator being capable of more sensitive adjustment.

The jig used in the graphite tests is a small laboratory apparatus, built by the manufacturers of the standard machine, for laboratory experiments. Plate II, *A*, shows the jig as set up for work. It comprises a small metal box, the bottom of which is made of a screen 3.25 by 2.5 inches. Air pressure of 2 to 4 ounces, furnished by a pressure blower or compressor is admitted to the bottom of the box under the screen through a small rapidly rotating valve. The rapid pulsation of the air, due to its passage through the valve, causes the material on the screen to remain in continuous agitation and partial suspension. Consequently, the coarse sandy impurities, being heavier, collect at the bottom, the lighter graphitic material being stratified above the sand. The jig is so arranged that operation is continuous, the sand being constantly collected and discharged from one side, while the lighter graphite is separated above the sand and discharged from the top of the column and on the other side.

Several tests were made with the jig, representative results being as follows:

Results of two representative tests with pneumatic jig.

	Carbon, per cent—	
	Sample 2.[a]	Sample 4.[b]
Heads	51.23	42.61
Concentrates	69.66	64.13
Tailings	15.11	3.14

It is seen that the recovery of clean tailings from the treatment of sample 4 was higher than from the treatment of sample 2. In order that the products after treatment can be compared with the original feed as to size and carbon content, the screen analysis and related data are given in Table 4 following:

---

[a] A calculated recovery of 91.25 per cent of the graphite in the jig concentrate is indicated.

[b] A calculated recovery of 97.5 per cent of the graphite in the jig concentrate is indicated.

TABLE 4.—*Results of screen analyses and of assays of jig concentrate and tailing.*

[Sample 4.]

Mesh.	Concentrate.				Tailing.			
	Proportion by weight.	Carbon assay.	Carbon, units.[a]	Total carbon.	Proportion by weight.	Carbon assay.	Carbon, units.[a]	Total carbon.
	Per cent.	*Per cent.*		*Per cent.*	*Per cent.*	*Per cent.*		*Per cent.*
14	None.	....	....	....	0.1	5.2	....	....
20	Tr.	....	....	....	.4	....	2.1	0.66
28	0.4	80.9	32.5	0.51	2.6	5.7	14.8	4.63
35	3.1	78.4	242.5	3.78	15.7	4.0	62.8	19.65
48	10.5	76.4	802.5	12.51	28.6	3.4	97.2	30.42
65	20.8	69.7	1,450	22.61	27.8	3.1	86.2	26.98
100	29.1	64.5	1,875	29.23	18.7	2.6	48.6	15.21
150	21.1	62.6	1,322	20.61	9.8	.8	7.8	2.44
200	5.8	55.1	319.5	4.98	.2	....	....	....
—200	9.3	39.8	370	5.77	.3	....	....	....
Total	100.1	64.12	6,414	100.00	99.2	3.19	....	....

[a] "Proportion by weight" times "carbon assay."

The results given in the table indicate that the air jig has practically the same field of application as the aspirator, also that it can be made to produce a cleaner tailing than the aspirator, but the aspirator will give a higher grade of finished product. This condition will not, however, interfere with the jig's usefulness, provided the material it removes from the crude concentrate is the coarse sand that causes the high loss of coarse flake in the buhr mill. An inspection of the various products of the jig will indicate that it does remove the coarse sand particles.

Many of the remarks regarding the use of the aspirator apply also to the air jig. There is no question that under certain circumstances the jig can be made to do valuable work in the preparation of crude graphite for crucible making, but the character of the crude concentrates themselves, and the other factors that enter into the commercial finishing of concentrates must be considered before the jig be recommended for use in preliminary treatment. If the jig is used only to do that work for which it is particularly adapted, the removal of coarse sand impurities, its success is assured.

TESTS WITH PEBBLE MILL.

Probably the most striking characteristics of graphite flakes are their toughness and shape. In these two respects the flakes differ greatly from the impurities usually associated with the graphite in the crude concentrates. It was hoped, therefore, that these properties could be utilized in separating the graphite from the associated impurities.

It was thought that the coarse sand impurities, although much harder than the graphite flakes, were so much more brittle that the concentrate could be so ground as to reduce materially the size of the sand particles without causing a material reduction in the size of the graphite particles. A careful survey of the different classes of grinding machinery indicated that this operation could be carried out with greatest chance of success in a mill of the ball or pebble type.

The difference in the specific gravities of the two materials was also a reason for believing that this sort of selective grinding could be done. It was thought that under the correct conditions of dilution the heavier sandy material would tend to classify in the bottom of the charge among the grinding balls where it would be ground more than the graphite, which, because of its lighter weight would tend to segregate higher in the charge and out of the grinding zone. Such a separation corresponds closely to that in a hydraulic classifier. The results obtained from experiments proved that in most particulars the assumptions were fundamentally correct.

After a treatment such as suggested, which would produce a product with the sandy constituents ground finer than the flake graphite, two methods of separating the sand and graphite are available.

One of the methods is by screening. Experiment showed that after such grinding, the grade of the original material could be raised 25 per cent or more by simply passing the ground concentrate over a 100-mesh screen. It will be recalled that 100-mesh material is the smallest size that can be satisfactorily used in crucible making.

The other method is by flotation, the process by which much of the crude concentrate produced in the Alabama field is made. Tests revealed that in order to recover the coarse flake in the ore by flotation, it was necessary to make a fairly low grade concentrate. The reason seemed to be that some of the silicious material was porous and sponge-like in structure, and contained quantities of entrained air which invariably caused the sand to float with the carbon.

Wet grinding of the concentrate in a ball mill tended to break up the pumice-like silica and to eliminate the entrapped air. When this had been done, the silica did not float as readily as before. Therefore, after grinding, the concentrate can be refloated and its grade raised to a degree corresponding to the amount of the spongy sand removed.

A small laboratory pebble mill (see Pl. I, *B*, p. 50) was used in the grinding tests. The charges consisted of 50 per cent water and 50 per cent crude concentrate. The grinding periods varied, but at first were of 30 minutes' duration.

A certain sample contained 90 per cent of the carbon as material coarser than 100 mesh which assayed 53.9 per cent carbon before grinding. After grinding in the pebble mill, 81 per cent of the carbon still remained on a 100-mesh screen as material which assayed

61.3 per cent carbon. These results indicated that a decided improvement had been made in the grade of the material coarser than 100-mesh, with a loss of only 9 per cent of the valuable coarse flake.

On the strength of these results, it was decided to make a series of tests under the conditions already described, but varying the length of the grinding period in each test. The results of this series of experiments are given in Table 5 following:

TABLE 5.—*Results of grinding graphite samples in pebble mill with equal parts of water.*

	Time of grinding (hours).	Grade of concentrate (percentage of carbon in +100-mesh material).	Carbon remaining on 100-mesh screen.	
			Percentage of total carbon of original sample.	Percentage of +100-mesh carbon in original sample.
Sample 2........	0	53.9	90.00	100.00
	¼	61.3	81.00	90.90
	½	77.6	80.60	89.60
	1	81.7	83.30	92.50
	1¼	79.8	81.40	90.40
	1½	81.4	72.60	80.60
	2	82.1	67.40	74.80
Sample 4........	0	47.9	80.5	100.00
	¼	70.6	65.9	81.80
	½	74.6	62.7	77.90
	1	81.4	62.5	77.60
	1¼	75.4	58.7	72.90
	1½	80.5	59.3	73.60
	2	82.4	51.1	63.50

The results are plotted in figures 16 and 17. The figures show that the correct time for grinding, under the conditions of this set of experiments, is the time that will produce the highest grade of +100-mesh material with a minimum destruction of the flake that originally existed in the concentrates coarser than 100 mesh.

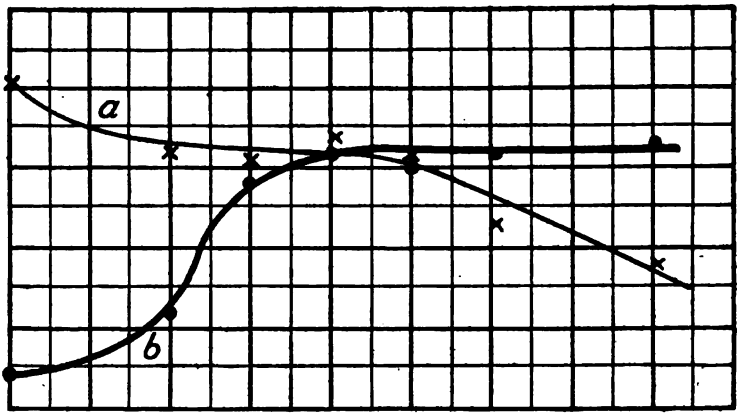


FIGURE 16.—Curves showing results of grinding sample 2 in pebble mill with equal parts of water. *a*, Recovery; *b*, carbon

On the curve this point will be at the intersection of the line representing the assay of the +100-mesh material with the line representing the recovery of +100-mesh carbon. For sample 2 the time is about one hour, whereas for sample 4 it is about 30 minutes.

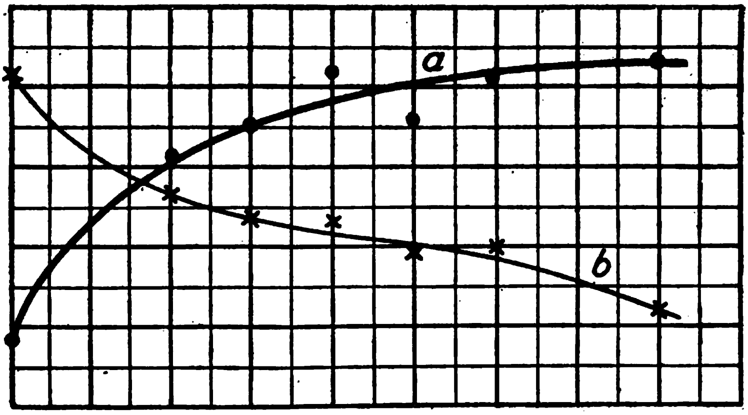


FIGURE 17.—Curves showing results of grinding sample 4 in pebble mill with equal parts of water. *a*, Carbon; *b*, recovery.

It is evident from these results that the conditions that give the best results will vary with the physical characteristics of the crude concentrate itself, and must be determined by actual experiment with the material that is to be treated in the commercial plant.

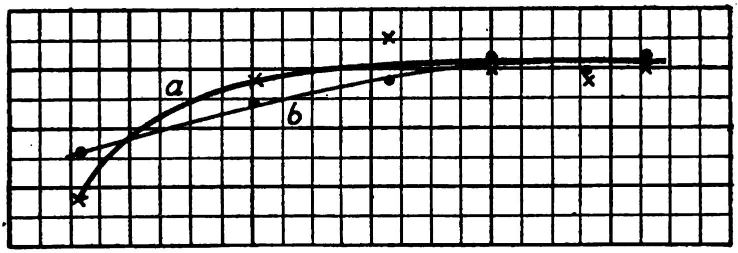


FIGURE 18.—Curves showing results of grinding sample 2 in a pebble mill with different percentages of water. *a*, Recovery; *b*, carbon.

In order to determine the effect of varying the amount of water in the pebble-mill charge, several tests were made, with sample 2, the other conditions being similar to those that had been proved to be the most satisfactory in the previous series. The results of these tests are shown in Table 6 following and in figure 18. The concentrates were fed to the mill at the rate of 400 grams per hour.

TABLE 6.—*Results of grinding graphite sample 2 in pebble mill with different percentages of water.*[a]

Quantity of water used (c. c.).	Grade of concentrate (percentage of carbon in +100-mesh material).	Carbon remaining on 100-mesh screen.		Water in mill pulp (per cent).
		Percentage of total carbon of original sample.	Percentage of +100-mesh carbon of original sample.	
0	53.9	90.00	100.00	0.0
300	77.6	73.7	81.90	42.8
400	81.7	83.3	92.50	50.0
500	83.5	87.0	96.60	55.55
600	85.0	84.50	93.30	60.0
700	84.1	83.40	92.50	63.6
800	84.7	85.10	94.50	66.6

[a] See also figure 18.

Figure 18 shows that no improvements in the results are to be noted after a dilution of 60 per cent water is reached. In this experiment the grade of the +100-mesh material was raised from 53.9 per cent carbon to 85 per cent carbon, with a loss of only 7 per cent of the carbon that was coarser than 100 mesh before grinding.

DISCUSSION OF PEBBLE-MILL RESULTS.

In summarizing the results obtained by the pebble-mill grinding, it may be said that the apparatus, when operated under the conditions that have been determined to be the most satisfactory, will raise the grade of the +100-mesh material in a crude concentrate to such a degree that the following buhr-mill grinding can be carried on to give the maximum results possible with it. At the same time, the results indicate that the pebble mill can not be used to produce a finished grade of product. In other words, the pebble-mill grinding should be used only as a step in the process, and preparatory to treatment in the buhr mill.

The choice of method of removing the impurities that are ground in the pebble mill will depend on the character of the concentrates that are being treated and other conditions that will be encountered in the commercial finishing plant.

The results obtained in the experiments here described indicate clearly that pebble-mill grinding has a wide field and can be made to produce favorable results even under the most adverse and difficult operating conditions.

TESTS WITH ELECTROSTATIC SEPARATOR.

The great difference between the electroconductivity of graphite and that of the principal impurities associated with it in the crude concentrates early suggested that a method of separation based on electroconductivity might be developed. Such a method has already been tried to a certain extent in the Alabama field, but the results in general have been unsatisfactory.

However, in one part of the Alabama district producing concentrates high in mica but very low in iron, the process worked well on a commercial scale and is being used there at the present time. Such material is particularly adapted for electrostatic treatment; hence the process gives satisfactory results, although certain changes in the separator as originally designed were made in order to overcome inherent mechanical defects. As this condition was only local, however, electrostatic separation was never successfully used in the rest of the district.

Improvements in the concentration mill made possible the production of concentrate carrying so much less iron that it was thought that the iron would no longer be a determining factor, and that hence the electrostatic process should offer much more chance for success than previously. Thus, in order to use this method of separation, it seemed necessary only to construct a machine that would eliminate the mechanical difficulties previously encountered in the operation of this type of apparatus.

With this end in view, a series of experiments was made with a large machine of the type at present on the market, in order to determine definitely the reason for its previous failure. However, with this machine the separation was not satisfactory. The poles of the machine are so arranged that the graphite particles receive a certain charge from one pole and are thrown into the field of the opposite pole; then the charge first obtained is neutralized and the particles are thrown in the direction of the first pole. Consequently the material progresses through the apparatus in a zigzag path, first in one direction and then in the opposite, with the result that no separation of the graphite from the nonelectroactive particles is obtained. The sand and other impurities, not being affected by the charges acting on the graphite, fall directly through the machine. Therefore, the graphite will be separated from the impurities while passing the first pole, but on passing the oppositely charged pole will be thrown in the opposite direction and into the sand from which it has just been separated. Eventually the mixture is discharged practically unchanged from its original state.

The experiments demonstrated that in order to overcome the faults mentioned it would be necessary so to design a machine that when a graphite particle had received a charge of a certain sign, it would carry this charge unneutralized, and not be forced in the opposite direction and lose its tendency to separate.

Many experiments were made with this end in view, and two types of apparatus were constructed. It was found possible to build a machine of such type that only one set of poles would tend to impart a charge to the graphite particles; these would be repelled directly into the concentrate hopper and given no chance to be neutralized and thrown back into the impurities.

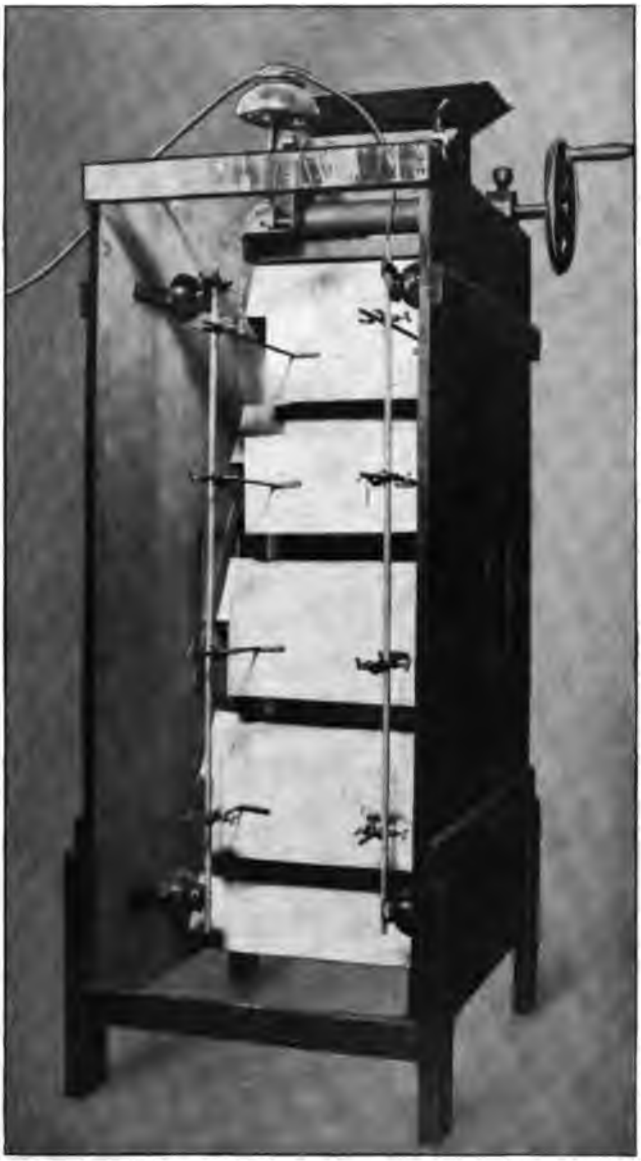
Figure 19 shows the principal details of the separator as finally constructed, and indicates the manner of its operation. A view of the separator as set up ready for work is shown in Plate III.

The material to be treated is placed in the hopper at the top of the apparatus and is fed from the hopper by a roller in the bottom. The graphite first strikes one of the inclined planes that carries a weak, negative charge. While the graphite is on this plane it therefore receives also a weak, negative charge. As the plane and the graphite both have the same charge, the graphite will be repelled and will jump from the negative vane into the field induced by the positive vanes. On coming into this opposite field, the graphite gives up its weak, negative charge and receives instead a much stronger positive one, which in turn causes it to be repelled violently from the positive plate and through the openings shown between the electrodes. After having passed to the back of the plates the graphite is free to fall directly into the concentrate hopper at the bottom of the separator.

The sand and mica, being much poorer conductors of electricity, receive such a weak charge from the negative plate on which they first fall that they are not repelled into the strong, positive field, but simply slide from one negative plate to the next and so on down through the machine until finally discharged into the tailings hopper at the bottom of the apparatus and in front of the concentrates hopper.

The electrical equipment used to operate the separator consisted of a 5-k. v. a. transformer designed and built as an oil-testing transformer. It is equipped with a hand-operated voltage regulator so arranged that the voltage can be varied from zero to the maximum. While the transformer has a secondary voltage of 50,000 volts, only half of this is available for rectification as the center of the secondary coil is permanently grounded to the transformer case.

The rectifier is of the mechanical type. It consists of a rotating disk driven by a synchronous motor. Stationary shoes and revolving electrodes on the disk make and break the circuit so as to rectify alternating waves. This arrangement gives a unidirection pulsating current necessary for the operation of the machine. Figure 20 shows the wiring diagram of this equipment.



ELECTROSTATIC GRAPHITE SEPARATOR.

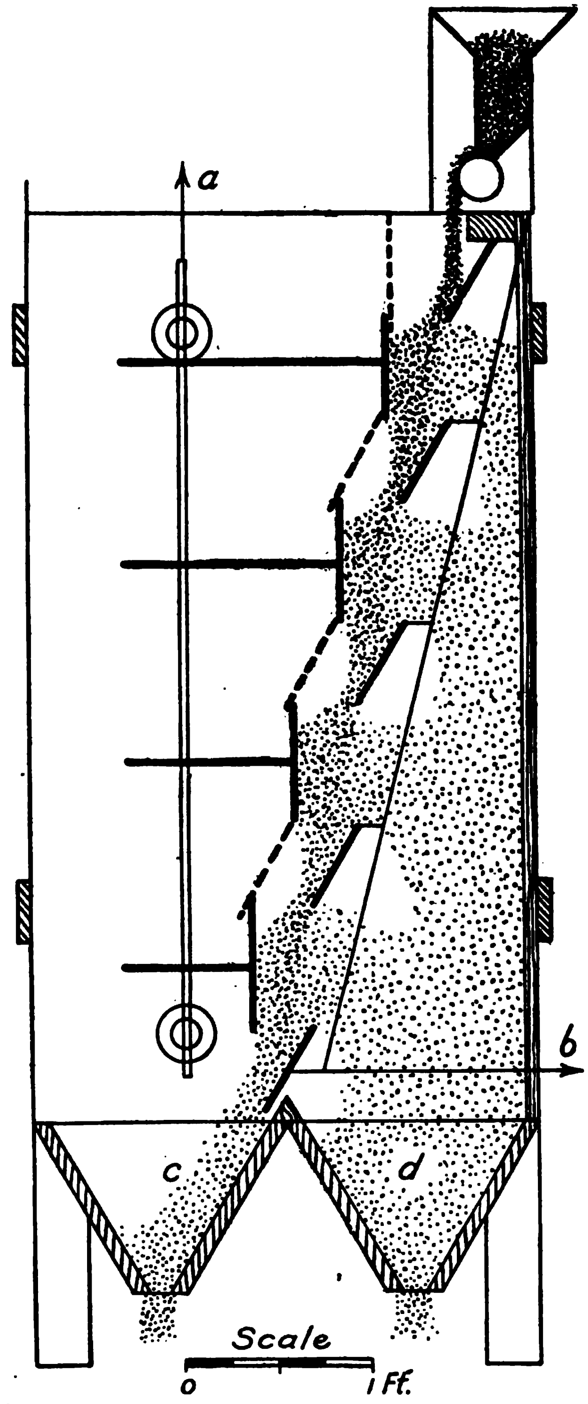


FIGURE 19.—Electrostatic graphite separator designed by Bureau of Mines. *a*, To rectifier; *b*, to ground; *c*, tailings; *d*, concentrate.

The table following gives a series of readings taken while the electrostatic separator was operated at the various voltages used in testing. This table shows that only 200 watts is required to operate the apparatus at the highest voltage. This is equivalent to only 0.87 k. v. a.

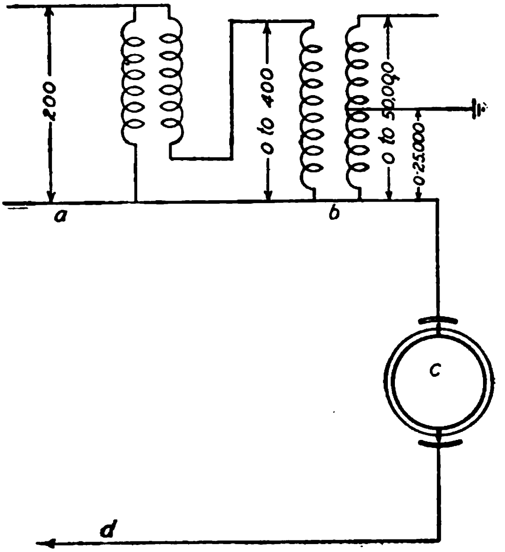


FIGURE 20.—Wiring diagram of electrical equipment used for electrostatic graphite separator designed by the Bureau of Mines.

Results of transformer readings taken in electrostatic-separator tests.

Primary coil.			Secondary coil.	
Volts.	Amperes.	Watts.	Power factor.	Volts.
446	2.22	2.12	0.21	27,900
440	1.95	2.00	.33	27,500
420	1.61	1.96	.29	26,250
388	1.15	1.52	.34	24,200
360	.90	1.32	.40	22,500
272	.50	.72	.53	17,000

The equipment (5 k. v. a.) is therefore large enough to operate a battery of six machines of the same type and size as the one described. Obviously the cost of power for the concentrating of graphite by electrostatic means will be small, an important consideration where power is costly.

After the completion of the equipment just described, a series of tests was made with the various concentrate samples in order to determine the efficiency of the device and the limitations of its use.

The voltage used in all of the tests was 28,500. The table following shows the results obtained in the treatment of the various samples.

Results of tests with electrostatic separator.

Sample No.	Percentage of graphite in—			Percentage of concentrates—	
	Feed.	Concentrates.	Tailing.	Coarser than 100 mesh.	Finer than 100 mesh.
1	48.95	59.02	25.16	61.5	24.2
2	51.25	74.23	13.90	75.8	59.8
2	51.23	78.81	19.04	80.2	61.6
4	42.61	68.58	8.30	71.1	24.7
4	42.61	75.89	....	80.6	65.2
2-100	81.2	87.62	....	88.5	60.4
Buhr mill:					
1	48.95	64.00	25.00	66.2	58.4
5	73.80	83.0	52.80	63.2	73.2
4	42.60	71.20	15.60	71.6	71.0
8	42.45	67.20	17.80	77.4	62.4
9	40.44	80.40	33.00	81.6	72.4

The results obtained with the electrostatic separator can be considered most satisfactory for two reasons. The tabulated results show that in every test the grade of the concentrates was considerably higher than the material fed to the machine. This improvement was accomplished without grinding, a step that will, in spite of all precautions, cause a certain reduction in size of the original coarse material in the crude concentrates.

In each of the eleven tests, except one, the concentrates were increased in grade by 10 to 40 per cent carbon. The proportion of carbon remaining in the tailings of the electrostatic separator varied greatly, but this variation was due, in almost every instance, to the carbon that is too fine to be acted upon by the apparatus in the most efficient manner, or to the variation in the number of flakes of graphite attached to pieces of sand large enough to cause them to be drawn into the tailings. Obviously the degree of variation will depend on the condition of the concentrates themselves and not on the treatment to which they are subjected. The photomicrographs presented in connection with the discussion of flake structure on pages 66 to 67, show this condition clearly.

As the value of graphite flake for crucible making depends on the assay of the material that is coarser than 100 mesh, the value of any plan of treatment will depend on the grade of this 100-mesh material produced. For this reason, the assays of the 100-mesh part of the concentrates are given, as well as the assays of the whole product. The results of the +100-mesh assays show that out of the eleven tests reported only two of the +100-mesh concentrates contained less than 70 per cent carbon, while seven of the number contained more than 75 per cent graphite. Such results were especially satisfactory in view of the fact that the concentrates had not been ground, with the consequent loss of coarse flake always resulting from a treatment of this nature.

It was found necessary to give the concentrates two treatments in the electrostatic separator in order to obtain optimum results. This requirement could be met in a commercial installation in one of two ways—by installing twice the number of separators required for a single treatment or by constructing apparatus containing twice the number of poles in the experimental equipment. It is probable that the latter method would prove the more satisfactory.

SUMMARY OF RESULTS WITH ELECTROSTATIC SEPARATOR.

The results obtained with the electrostatic separator prove conclusively that electrostatic separation can be successfully used in the finishing of graphite. They also indicate that the separators on the market at present will have to be changed in certain respects in order to make possible the obtaining of satisfactory results by their use. The results also seem to prove that the apparatus has a wider application than is possible with any of the other devices tested, with the possible exception of the buhr mill.

The electrostatic process seems also to be the most economical, as the cost of power is low and the loss of coarse and valuable flake is small.

At the same time, it is to be expected that, although the device will invariably improve the grade of the concentrates treated, it will not yield a finished product containing 85 per cent carbon, which is satisfactory for crucible manfacture.

However, the improvement in grade of the concentrates tested was so great as to indicate that electrostatic separation will, with few exceptions, produce a feed for the buhr mill that will be ideal for that apparatus to finish to the crucible requirements without excessive loss of coarse flake. The removal of the coarse sand was almost complete.

The results already given show that although the electrostatic separator will benefit the concentrates the degree of beneficiation will vary. Seemingly, the degree of variation depends on the physical condition of the concentrates themselves previous to treatment. In other words, the results to be obtained with this separator, like those with any other type of equipment, will depend on the concentrates themselves. Hence there can be no doubt that conditions will sometimes be met under which this type of treatment would not be as satisfactory as some other that might be developed for the removal of the coarse sand. However, the process has a more general application than any of the methods described thus far.

FLOTATION AS A FINISHING PROCESS.

Flotation as a process of concentrating the graphitic content of ores has been used for some time and with a fair degree of success. For this reason and because graphite has a much greater tendency to float than the impurities that are usually found associated with it the idea of refloating the concentrate to remove the impurities before buhr-mill treatment was suggested.

There are two primary reasons for thinking that double flotation could be used in this connection. The first is the law of concentration that the grade of the concentrates produced by a concentration process will vary directly with the richness of the feed being treated. As it is possible to produce a 45 per cent graphite concentrate from ore carrying only 3 per cent graphite, it is reasonable to believe that, under the proper conditions, it should be feasible to make a concentrate carrying, say 70 per cent or more of graphite from a feed carrying 40 per cent or more of graphite.

The other possible reason for the success of flotation has already been suggested in the discussion of the use of the pebble mill. The point in mind is the porous condition of much of the silicious material that is found in the crude flotation concentrates. It was believed that if the concentrates from the first flotation treatment were to be ground or given some other treatment by which the sand could be reduced in size sufficiently to liberate the entrained air this siliceous material could be largely removed by a second flotation treatment.

FLOTATION TESTS.

In order to prove or disprove these assumptions, three tests were made with a sample of flotation concentrate. One of these tests was made after the sample had been ground in the buhr mill, one after a treatment in the pebble mill, and one with material just as received without any treatment preliminary to the refloating.

The results obtained by this series of tests were as follows:

Results obtained in three reflotation tests.

Sample No.	Previous treatment before refloating.	Graphite in—			Recovery.
		Heads.	Concentrates.	Tails.	
		Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
2	None	51.23	67.35	10.8	93.7
2	Buhr mill	51.23	80.73	2.1	98.5
2	Pebble mill	51.23	83.40	5.6	97.7

These results make it evident that simple reflotation, without a preliminary treatment, will materially raise the grade of the product, and give a high recovery of the carbon content of the feed. However, as would be expected, the results do not indicate that simple reflotation would be satisfactory in commercial practice.

As regards reflotation following treatment in buhr or pebble mills, the results are such as to justify its adoption in a commercial installation.

Both the mechanical and the pneumatic types of flotation machines were used in the tests and, as far as could be noted, neither of the machines showed an advantage over the other.

Several oil mixtures were used. A mixture of 2 pounds of No. 17 oil and 0.75 pound of No. 5 oil, both made by the General Naval Stores Co., was found to be satisfactory in each test.

A series of tests was also made with sample 2 in order to determine the different results that could be obtained with different oil mixtures and various periods of agitation. These results are added for general interest, and to show what poor results can be obtained from a sample, by inefficient operation and incorrect conditions, as compared with the satisfactory results possible under proper conditions.

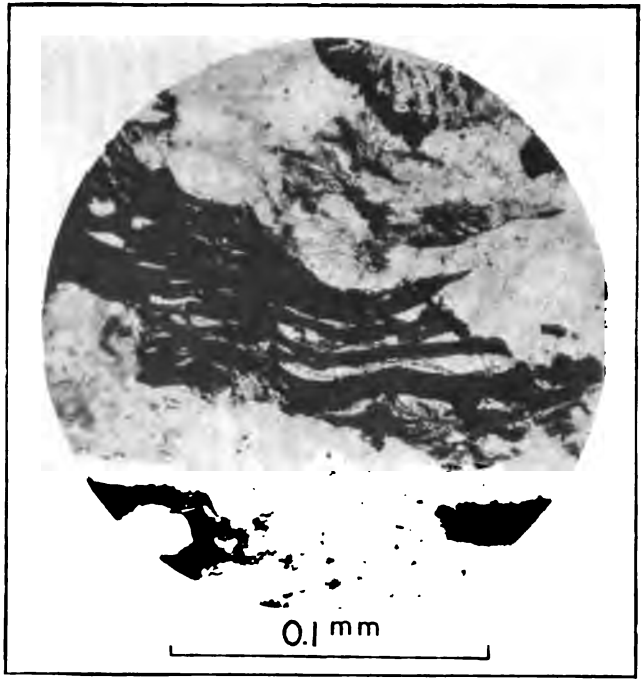
Results of refloating crude concentrates.

Sample No.	Preliminary treatment.	Percentage of graphite in—			Assay of concentrates.	
		Heads.	Concentrates.	Tails.	Coarser than 100 mesh.	Finer then 100 mesh.
2	Buhr mill	51.23	84.24	39.3	88.00	70.5
4	do	42.61	69.69	2.5	73.9	63.1
2	do	51.23	80.73	2.1	82.7	67.6
2	Pebble mill	51.23	83.10	....	86.0	77.1
2	None	51.23	67.35	10.8	68.4	50.3
2	Pebble mill	51.23	87.6	7.5	....	....
2	do	51.23	76.27	17.68	78.6	49.0
2	do	51.23	83.4	5.6	....	....

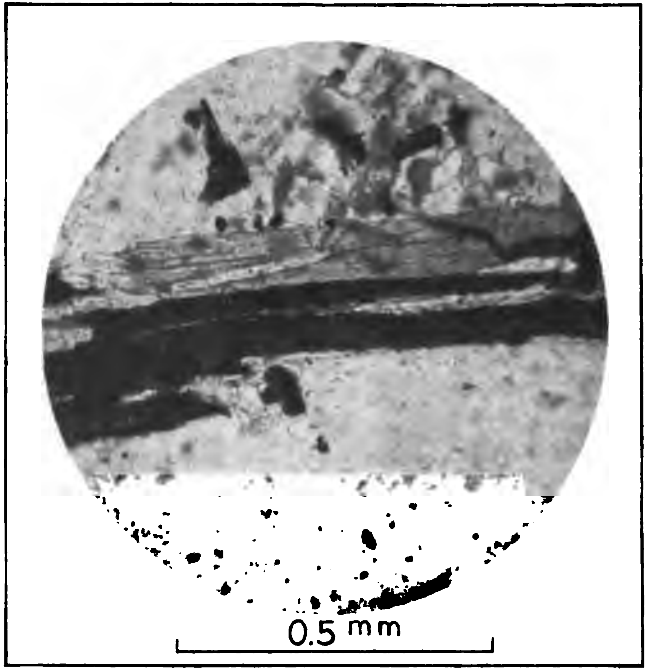
In discussing the use of flotation, it might be said that the process when used to treat concentrates that have been previously ground, for instance in a pebble mill, will produce a material that will be particularly amenable to the final treatment in a buhr mill. The results indicate that supplementary treatment will be necessary to produce material containing the required 90 per cent graphite.

THE BUHR MILL; ITS PLACE IN GRAPHITE FINISHING.

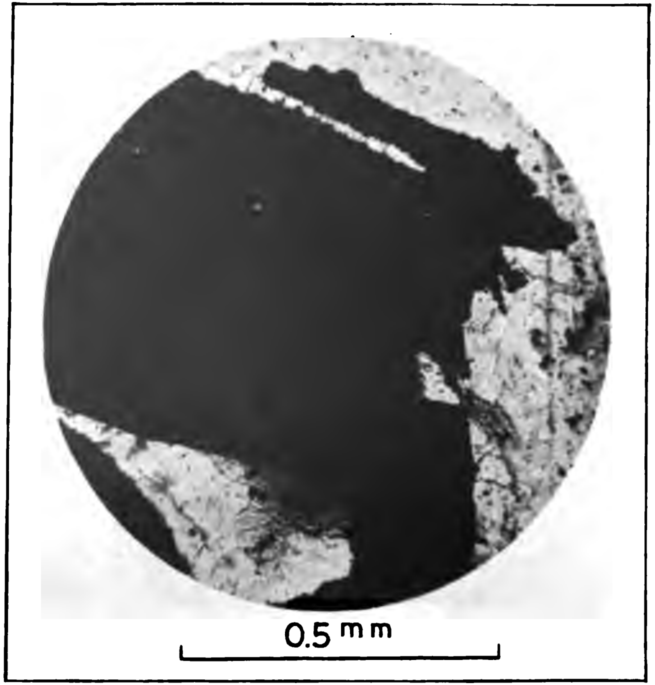
The function of the buhr mill has been previously discussed in this report. The action of the machine has also been suggested, with the two principal reasons for unsatisfactory results under certain conditions, namely, lack of uniformity in the product finished by it, and the excessive destruction of flake sometimes attending its use.



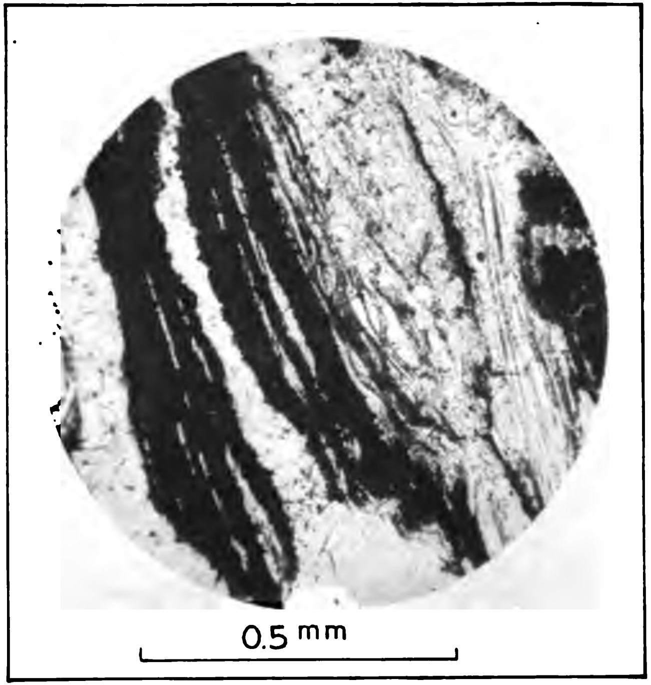
A. MICROPHOTOGRAPH OF GRAPHITE ORE, SHOWING IRREGULAR STRUCTURE.



B. MICROPHOTOGRAPH OF GRAPHITE ORE, SHOWING BANDED STRUCTURE.



A. MICROPHOTOGRAPH OF GRAPHITE ORE, SHOWING A LARGE PLATE WITH A TRIANGULAR INCLUSION.



B. MICROPHOTOGRAPH OF GRAPHITE ORE, SHOWING IMPURITIES EMBEDDED IN FLAKES.

During the comparatively long period that this machine has been used to finish graphite, methods of overcoming inherent difficulties in its use have become evident. One requirement is that the concentrate be of comparatively high grade before grinding in the buhr mill. With such material, no difficulty is encountered in consistently producing a crucible stock assaying 90 per cent graphite, provided the other conditions are correct.

In order that the excessive destruction of coarse flake incident to buhr mill grinding may be eliminated, it is necessary only to remove the coarse, gritty particles, or at least those coarse enough to require a large amount of grinding in the buhr mill.

Stated simply, when the buhr mill is used principally as a grinding machine on low-grade material, the grade of the product resulting can not be depended upon, and an excessively large proportion of the flake will be ground to dust. When, on the other hand, the machine is used only as a polishing or buffing machine, it can be trusted to give satisfactory results.

As already mentioned, the limitations of the mill were realized early in the progress of the work, and the experiments were conducted accordingly. Most of the products resulting from the various treatments contained less than the required 90 per cent of carbon and, therefore, could be used by the crucible makers only after a treatment in the buhr mill, to bring them to the necessary grade.

A view of the buhr mill used in the experimental work is shown in Plate II, *B* (p. 53).

RELATION OF FLAKE STRUCTURE TO FINISHING.

In order to show more clearly the truth of the assumptions previously stated, microscopic studies were made of graphite flakes, both before and after finishing. Also a series of microphotographs were made of this material. Four of these are reproduced in Plates IV and V.

The photographs show that the flakes were composed of series of thin laminations or plates of graphite, interbedded with thin pieces of quartz, mica, and other impurities. In some material these impurities were seen to be entirely surrounded by the carbon.

In order to determine the proportion of the impurities held in this manner, flakes of seemingly pure graphite, as it occurs naturally in the ore, were carefully picked by hand from the crude concentrate and assayed. In selecting the sample, special care was taken that nothing but the free, natural flakes were obtained for assay. The sample obtained in this manner was then carefully assayed, with the following results: Moisture, 1.26 per cent; carbon, 90.2 per cent; ash (impurities), 8.54 per cent. Examination of the ash under the microscope showed that it consisted principally of mica with some quartz. This experiment indicated conclusively that impurities are imbedded between the laminations of the graphite, and also that the removal of all impurities not attached to the graphite flakes would give a finished product assaying only 90 per cent carbon.

This figure is, then, close to the limit of graphitic content possible in a finished material, even after treatment by a perfect finishing process.

Evidently the interlaminated impurities must be freed from the graphite flakes before they can be removed from the crude concentrate, and the only feasible method of accomplishing this is by separation of the graphite flakes into their individual laminations. It follows that the more there are of these interbedded layers to be removed, the greater will be the number of plates into which the flakes must be subdivided. Also, the greater the number of plates made in the treatment, the thinner they must be and, hence, the smaller the average diameter will tend to be.

We have seen that the value of the graphite for crucible manufacture depends largely on the diameter of the flakes composing the crucible stock. Therefore, if all of the interbedded impurities are to be removed, the resulting flakes will be so thin and of such small diameter that the value of the graphite for crucible making will be lessened.

Theoretically it would be possible so to separate the flakes into individual plates by buhr-mill grinding as to make a finished product in such a fine state of division that it will be valueless. Hence, the degree to which the finishing can be accomplished in a commercial plant will depend on the number and the characteristics of the interbedded impurities in the crude flakes. These factors will also determine the amount of grinding that is permissible in the buhr mill to permit removal of impurities, and will determine the grade of finished product that will be produced by a definite amount of such grinding. The proportion of dust produced will also depend on the same factors.

In other words, the interbedded material is the important factor in determining the economic possibilities of the finishing process. If the flake graphite contained in a certain crude concentrate is of such a nature that, in order to produce 90 per cent carbon in the finished product, an undue proportion of the carbon coarser than 100 mesh is destroyed, obviously the product will not be satisfactory.

There is no doubt that the buhr mill is the best commercial apparatus that has been developed to date for separating the flakes of graphite into their individual laminations and at the same time reducing the freed impurities to a size permitting their removal by screening or bolting.

Several important features must be considered in grinding graphite concentrates in a buhr mill. The first point to be emphasized is that in order to produce a good crucible stock it is necessary to do more than remove the gangue present in the crude concentrates; it is also imperative that impurities interfoliated between the graphite laminations be removed. Clearly, in order to remove such impurities, some sort of grinding or rubbing process must be used, and, preferably, this should follow the removal of superficial or incidental impurities, so that these can not act as abrasive on the graphite flakes during the grinding.

From a survey of the grinding or polishing apparatus available, the conclusion is reached that the buhr mill is peculiarly adapted for this work, and that it is the only machine that can be depended on to do the required work in a satisfactory manner.

The results of the experiments indicate that the efficiency of the buhr mill, and hence of the whole finishing process, will depend on the character of the material that is being ground, and this in turn will depend both on the character of the flakes as they occur in the ore and on their treatment previous to grinding in the buhr mill. No hard and fast rules can be prescribed for the treatment of any particular concentrate, but in order to determine the most efficient treatment, it will be necessary to ascertain by experiment the best system to be followed.

The discussion indicates that satisfactory results depend on so many and such varied factors that undoubtedly there are certain concentrates produced from particular ore that can not be made into a crucible stock at a profit. Hence, it is true that although the equipment, the process, and the skill used in operation are all important for successful results, there is the added necessity that the flake itself meet certain requirements.

IMPURITIES IN GRAPHITE.

In order to learn more of the chemical nature of the impurities that were encountered in the crude concentrates, and in the finished graphitic material, chemical analyses were made of ash samples from some of the representative graphite samples. The results are presented in the table following.

Results of analyses of samples of graphite ash.

Constituent.	Ash from graphite sample 2.	Ash from graphite sample 4.	Ash from sample 2 after special treatment.[a]	Ash from sample 2 after special treatment.[b]
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
SiO_2	65.80	78.71	45.46	51.80
Al_2O_3	20.53	11.45	36.90	33.75
Fe_2O_3	10.77	8.65	14.00	11.85
CaO	.80	.73	.75	.67
Na_2O and K_2O	2.46	.71	2.68	2.54
Total	100.36	100.25	99.79	99.91

[a] Treated in electrostatic separator and in buhr mill.
[b] Treated in buhr mill, given flotation treatment, and again put through buhr mill.

These results indicate that the finishing process tends to remove the silica but causes the alumina to be concentrated. This is, of course, due to the removal of the silica in a greater proportion than the mica, which contains the greater part of the aluminum oxide. This result is to be expected for the reason that the silica can be more easily ground because of its more massive form and brittle character.

It should be mentioned that mica, because of its flat shape and its toughness, is probably the most difficult impurity to remove in graphite refining.

RESULTS FROM COMBINATION OF VARIOUS TREATMENTS.

After considering the various apparatus that might be used in the production of crucible stock from crude concentrates, and a few of the factors that might be expected to affect the results, experiments were made to determine the results that could be obtained by logical combinations of the various schemes and apparatus.

The results already obtained indicated that two general schemes could be developed for a complete finishing process to produce crucible stock from Alabama concentrates. One of these schemes may be termed a wet process and the other a dry process.

The dry process is rather simple and involves only a treatment of the crude concentrate in the electrostatic separator, followed by grinding in the buhr mill and screening. The coarse sizes from the screen constitute the finished graphite suitable for crucible manufacture and these were found to carry 90 per cent carbon and flakes of the correct size for crucible manufacture.

The wet process is also simple, though involving one more step than the dry process. Wet concentrates are used in each step of this process, except that in the last step, grinding in the buhr mill, dry concentrates are used. Thus, the wet process should offer some advantages over the dry process. The wet concentrates are ground in the pebble mill for a period of time previously determined to be correct. The ground material is then floated in a standard flotation machine. The concentrates produced are dried and reground in the buhr mill, after which they are screened. The screen used is 100 mesh and the oversize is the finished crucible stock.

A test of the dry process was made with sample 2, which assayed 51.23 per cent carbon. The material was twice treated in the electrostatic separator, a small sample was cut from the concentrates for analysis, and the remainder ground in the buhr mill. The product from this grinding was then screened and assayed. The whole product from the electrostatic treatment assayed 74.23 per cent carbon; the +100-mesh material contained 75.8 per cent and the —100-mesh substance, 59.8 per cent. The whole product from the grinding in the buhr mill assayed 78 per cent carbon; the —100-mesh material showed 45 per cent carbon, and the +100-mesh product, or crucible stock, 90.5 per cent. The total recovery of graphite in the finished crucible stock was 83 per cent of the graphite in the original sample.

The product obtained by grinding the concentrate from the electrostatic separator in the buhr mill was submitted to screen analysis and assay with the following results:

Results of use of electrostatic separator and of buhr mill.

Mesh.	Percentage of each size, by weight.	Carbon assay. (per cent).	Units of carbon, each size.[a]	Percentage of total carbon.[b]
+14	Tr.	....	....	....
+20	Tr.	....	....	....
28	0.9	92.8	83.00	1.06
35	5.1	91.8	468.00	6.00
48	15.2	91.4	1,388.00	17.80
65	23.0	90.1	2,054.00	26.30
100	26.3	86.2	2,265.00	29.00
150	13.8	85.6	1,181.00	15.10
200	4.3	39.8	171.00	2.20
—200	10.8	17.4	188.00	2.41
Total	99.4	78.4	7,798.00	99.87

[a] Product of column 2 times column 3.
[b] Results obtained by dividing figures in column 4 by sum total of column 4.

The following tabulation shows calculated results based on the foregoing data and also the results of actual analyses:

Selected data showing results of use of electrostatic separator and of buhr mill.

Material.	Percentage of final product.	Carbon assay (per cent).	Units of carbon.[a]	Percentage of total carbon.[b]
Calculated results:				
Crucible stock (material over 100 mesh)	70.5	88.8	6,258.0	80.2
Dust (material less than 100 mesh)	28.9	53.3	1,540.0	19.8
Total	99.4	78.4	7,798.0	100.0
Results of analyses:				
Crucible stock	70.5	90.5	6,380.2	83.1
Dust	28.9	45.0	1,300.5	16.9
Total	99.4	77.2	7,680.7	100.0

[a] Column 2 times column 3.
[b] Results obtained by dividing figures in column 4 by sum total of column 4.

These results were exceptionally satisfactory. The grade of the coarse material was higher than the 90 per cent product required by the crucible makers, whereas the loss of less than 20 per cent of the total graphite in the original sample was lower than the loss in the finishing mills that are producing graphite of crucible grade from the Alabama concentrates.

As already mentioned, the whole success of a finishing process depends on the removal of a large percentage of coarse hard impurities that tend to cause excessive destruction of valuable flake in the buhr mill grinding. Such removal is, of course, the function of the electrostatic treatment, and the results prove how satisfactory such treatment was.

A test of the wet method was next made with 400 grams of sample 2 concentrate. The material was ground with 600 c. c. of water for one hour and floated in the Callow pneumatic machine. The concentrate from this treatment, when filtered and dried, contained 83 per cent carbon. This concentrate was put through the buhr mill, when the +100-mesh material was found to contain 90.3 per cent carbon. The recovery of the graphite was 80.2 per cent of the carbon in the original sample. Tabulated results follow.

Results of treating concentrates in pebble mill, flotation machine, and buhr mill.

Mesh.	Percentage, by weight, of each size.	Carbon assay (per cent).	Units of carbon, each size.[a]	Percentage of total carbon.[b]
14	None.			
20				
28	1.2	88.6	106.00	1.29
35	5.9	94.0	554.00	6.70
48	16.0	90.8	1,455.00	17.61
65	24.8	93.0	2,305.00	27.94
100	26.6	89.0	2,365.00	28.65
150	12.9	83.4	1,076.00	13.01
200	13.5	63.6	223.00	2.70
−200	10.1	17.4	167.00	2.02
Total	100.0	82.5	8,251.00	99.91

[a] Column 2 by column 3.
[b] Results obtained by dividing figures in column 4 by sum total of column 4.

The following tabulation shows results based on the foregoing data:

Selected data showing results of use of pebble mill, flotation machine, and buhr mill.

Material.	Percentage of final products.	Carbon assay (per cent).	Units of carbon, each size.[a]	Percentage of original carbon.[b]
Crucible stock (material over 100 mesh)	73.5	91.0	6,690.0	81.5
Dust (material less than 100 mesh)	26.5	57.5	1,525.0	18.5
Total	100.0	82.5	8,215.0	100.0

[a] Column 2 times column 3.
[b] Results obtained by dividing figures in column 4 by sum total of column 4.

As with the dry process, the success of this treatment depends on the removal of the coarse impurities before the final buhr-mill grinding, and such removal is accomplished by the grinding in the pebble mill followed by flotation.

Owing to the short time available for the graphite investigation it was impossible to test all the samples with the combination treatment, but there can be no doubt that the results of such treatment would check closely with the results already described.

GENERAL SUMMARY AND DISCUSSION OF RESULTS.

As a result of the investigations made to determine the most satisfactory methods of refining crude graphite concentrates, several important facts were developed.

Probably the most important of these is that each concentrate to be treated is a problem in itself, hence no treatment applicable to all kinds of concentrate can be prescribed. Such a limitation is due to the differences between the characteristics of the crude graphite flakes and those of their associated impurities. The most important characteristics that will affect the type of treatment to be applied are the hardness, toughness, diameter, and thickness of the flakes and their contents of interfoliated impurities. The different physical properties affect the amount and intensity of the grinding necessary to remove the impurities without the destruction of too large a proportional amount of the large and more valuable flakes.

Another important factor in determining a satisfactory finishing process is the kind of impurities that must be removed. When these impurities exist free from the flakes of carbon they can be removed, to a great degree, with a small loss of coarse carbon. When these impurities are soft and of small size they can be removed after a slight and nondestructive grinding operation, which will in all probability cause a comparatively small loss of valuable flake.

On the other hand, coarse, hard impurities are much more difficult to handle in a satisfactory manner. This type of material offers the greatest problem to the operator of the finishing mill. The results obtained in the experiments indicate that the success of the finishing process will depend on the success obtained in the removal of this sort of impurities at the earlier stages of the finishing operations.

The investigations have also brought out the fact that the buhr mill is a necessary apparatus in all processes for graphite finishing. The buhr mill separates the flakes into their constituent laminations and prepares them for the removal of the interbedded or interfoliated impurities.

The aspirator and the pneumatic jig were found to have an application under certain circumstances. It was seen, however, that such apparatus can be considered only for preliminary treatment previous to the regular finishing process and should be used only for the removal of very coarse, hard, free impurities that are encountered from time to time.

The experiments proved fairly conclusively that the use of either the electrostatic separator or the pebble mill, followed by flotation, will be applicable to all types of concentrates produced in the Alabama fields. The material produced by either of these types of apparatus can be ground in the buhr mill with satisfactory results.

The different processes of finishing graphite may be classified under two general heads—wet processes and dry processes. The decision as to which of the two should be used is dependent on the characteristics of the concentrates themselves. It is important that neither be adopted except after the necessary experiments and on the advice of an engineer thoroughly conversant with the problems to be encountered in the treatment of the particular concentrate in mind and with the economic conditions that will exist in the plant.

It can be said that with rare exceptions a satisfactory crucible stock can be produced from all of the concentrates that are at present made from the Alabama ores, and that the recovery of +100-mesh flake at the majority of finishing plants now operating can probably be improved. It should be possible to keep the recovery of material coarser than 100 mesh at 70 per cent of the total carbon in the original concentrates, and in many instances this can be increased to 80 per cent or more. These figures are sufficiently high to make profitable many of the plants that are not now operated except at a loss.

In conclusion, it should be emphasized that the results obtained in finishing the crude graphite concentrates are dependent largely on the character of the crude flake itself as contained in the ores. Moreover, mills that are producing a concentrate containing large tough flake in their concentrating plants, can also produce a satisfactory crucible stock at a profit from a finishing plant which has been correctly designed and intelligently managed. The operator who is unfortunate in having to mine small, low-grade flake requiring a large amount of intensive grinding, and which is associated with a large quantity of coarse siliceous or micaceous impurities, is bound to have difficulty in producing the required grade of crucible stock at a profit.

As in all mining ventures, success or failure depends more on the character of the raw ore than on the treatment given the ore after it has been mined.

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DEPARTMENT OF THE INTERIOR
FRANKLIN K. LANE, SECRETARY
BUREAU OF MINES
VAN. H. MANNING, DIRECTOR

ABSTRACTS OF CURRENT DECISIONS
ON
MINES AND MINING

REPORTED FROM
MAY TO SEPTEMBER, 1915

BY

J. W. THOMPSON

WASHINGTON
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PREFACE.

This bulletin is the fifth of its kind to be published by the Bureau of Mines, the four preceding being Bulletins 61, 79, 90, and 101.

The wide demand for the information contained in these bulletins has led the bureau to decide to issue similar bulletins with sufficient frequency to keep reasonably current the records of decisions of Federal and State courts of last resort on questions relating to the mineral industry.

The bureau will gladly welcome and consider any suggestions looking to improvement in the matter contained in these bulletins or the manner in which it is presented. The purpose of the bulletins will continue to be to improve directly or indirectly mining conditions and to promote the health and safety of miners by the prompt publication of decisions, and to this end it is desired that the bulletins reach all persons who are interested.

VAN. H. MANNING.

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ABSTRACTS OF CURRENT DECISIONS ON MINES AND MINING, REPORTED FROM MAY TO SEPTEMBER, 1915.

By J. W. Thompson.

MINERALS AND MINERAL LANDS.

MINERALS.

OIL AND GAS AS MINERALS.

Oil and gas within the ground are minerals and the fact that they have attributes not common to other minerals because of their fugitive nature or vagrant habit, and the disposition to percolate, and the possibility of their escape from beneath one part of the surface to another, does not remove them from the class of minerals.

Texas Co. *v.* Daugherty (Texas), 176 Southwestern, 717, p. 719, May, 1915.

OWNERSHIP OF TAILINGS—ABANDONMENT.

A corporation engaged in milling and the reduction of ores deposited the tailings therefrom upon a portion of its own land lying in a gulch through which water flows at times in great volumes and with great force and notwithstanding all reasonable efforts by dams and otherwise to retain its tailings, the tailings eventually were forced upon the lands of an adjoining owner; these tailings were valuable and their retreatment, intended by the mill owner, profitable; and the fact that the tailings were so washed down upon the lands of the adjoining owner and permitted to accumulate and remain for some considerable time is not sufficient to show an abandonment on the part of the owner of the tailings, where it is clear that the owner had no intention of so doing, but on the contrary, always intended to conserve and retreat the tailings.

Goldfield Consolidated Milling, etc., Co. *v.* Old Sandstrom Annex Gold Mining Co. (Nevada), 150 Pacific, 313, p. 317, July, 1915.

SALE AND CONVEYANCE.

CONSTRUCTION OF DEED.

All parts of a deed conveying mining property must be construed together without regard to its mere formal divisions.

Brier Hill Collieries *v.* Gernt (Tennessee), 175 Southwestern, 560, p. 561, January, 1915.

CONSTRUCTION OF DEED—CONVEYANCE LIMITED TO INTEREST OF GRANTOR.

A deed conveying certain described mineral lands and providing that the grantors convey and assign all their right, title, claim, and interest in and to the property described as fully as the same is in them vested, and not otherwise, limits the previous conveying words and confines and restricts them to such right, title, and interest only in the lands described as was at the time vested in the vendors; and such a deed does not purport to convey, and does not convey, a tract of land previously conveyed to another person, though included within the description contained in the deed, and the conveyance in such case does not operate even as color of title to such prior conveyed tract.

Brier Hill Collieries *v.* Gernt (Tennessee), 175 Southwestern, 560, p. 561, January, 1915.

SALE OF SURFACE—RESERVATION OF EASEMENT—CONSTRUCTION OF DEED.

A deed conveying the surface of certain mineral lands described, with a reservation to the grantor of minerals and the usual mining rights and certain parts of a river and creek bank, together with a "right to build railroads through said lands in order to reach other lands beyond and above," is not to be limited to the lands then owned by the grantor, as distinguished from those that he might afterwards acquire, where it appears that the grantor was engaged in acquiring mineral lands and in bodying them up so as to make profitable mining propositions, and in such case it will be presumed that the grantor reserved the rights described in the deed in his own interest and in that of his heirs and assigns and for the purposes of the occupation and projects in which he was then engaged; and it would appear to be equally advantageous to reserve the right to build railroads to reach lands to which the grantor never got title, as it might be impossible for him to utilize, either by mining or sale, his lands except in connection with the adjoining lands of others, and it is reasonable that the reservation was intended to cover such lands, as it was probable that the grantor or his heirs might be interested in having served by the railroad, in connection with his own lands "above and beyond" the lands described; and it seems reasonable that the grantor intended the reservation to be as broad as the necessity, and if the necessity included other lands than those then owned by him or those thereafter acquired by him, it should be held to extend to and cover such other lands.

Oak Leaf Coal Co., In re, 225 Federal, 126, p. 127, July, 1915.

CONTRACT OF PURCHASE—CONSIDERATION—ARBITRARY PRICE.

A mining corporation whose specific operations have failed and its plant has been destroyed, its mines closed and flooded, and its operations expensive and unprofitable for a long period of years, can not be compelled to borrow unnecessarily large sums of money to pay its debts and expenses, or to enable it to rehabilitate its mines and experiment and explore unworkable bodies of ore; and under such conditions there is common law power, coextensive with any possibly given by statutes or articles, and without unanimous consent of the stockholders to sell the property of the corporation and the fact that it has large bodies of unworkable zinc and silver ore, and much virgin ground will not make the sale at any arbitrary price necessarily illegal where there is no market value for such properties and where it is impossible to ascertain an accurate value or a certain basis for ascertaining the value of its properties.

Geddes *v.* Anaconda Copper Min. Co., 222 Federal, 129, p. 131, May, 1915.

CONTRACT OF PURCHASE—EFFECT AND QUIETING TITLE.

A contract which transfers and assigns an estate in mining property and at the same time withholds from the transferee or assignee all right of control over such property, is lacking in the elements of a sale or transfer of title, and vests no rights of ownership or title in the transferee, for the reason that a person who has no right to control, handle, or dispose of a mine can not be considered its owner, as the essential attributes of ownership of property are the rights in the owner to control, handle, and dispose of the thing owned; and in an action to quiet title to such mining property such a contract is no evidence of title and will not authorize a judgment quieting title in the mining property to the promisee.

Hardinge *v.* Empire Zinc Co. (Arizona), 148 Pacific, 306, p. 312, May, 1915.

CONTRACT FOR UNDIVIDED INTEREST—RESERVING CONTROL AND DISPOSAL.

A mining corporation holding options to lease and purchase mining property, transferred and assigned, by the officers and persons in whose names the contract was made, an undivided one-twentieth interest in the mining properties described to a certain named stockholder, subject to the control, handling, and disposal by the officers and persons in whose names the contract was executed, as in their judgment shall seem best for all concerned. This limiting clause was understood and construed by the assignee to the effect that he had no control of his interest but that such interest was subject to the control of the persons named and with power to sell and dispose of the same at the same rate and on the same conditions that they should sell their own, and the contract thus construed and the words of the contract all considered make clear that it was the purpose and intention of the parties to secure to such assignee a one-twentieth interest of the property asset, and there was no intention of conveying to him any interest in the title to such asset, the mining property itself, and the purpose was to withhold from such assignee the right to control and dispose of his interest and not to make the contract in effect a conveyance of title to the property described. And under this contract the assignee could not maintain an action to quiet the title to his undivided interest.

Hardinge *v.* Empire Zinc Co. (Arizona), 148 Pacific, 306, p. 310, May, 1915.

CONTRACT FOR SALE OF MINERAL LANDS—RELIEF.

Equity will not entertain a suit to cancel a contract for the sale of a large tract of mineral lands, alleged to be valuable for mining purposes, on the ground that it was procured by fraudulent representations or on the ground that such an executory contract is a cloud upon the complainant's title, as in such case the complainant has a full, adequate, and complete remedy at law, either by action or defense, as the contract involved is simply executory.

Big Huff Coal Co. *v.* Thomas (West Virginia), 85 Southeastern, 171, p. 173, April, 1915.

CONTRACT OF PURCHASE—CONSTRUCTION AND EFFECT.

The execution of a written instrument by the owner of certain described mining land whereby he offered and agreed to sell to the person named therein for a stated consideration a certain part to be paid in cash as soon as an engineer's report upon the property was completed and abstracts of title to the property had been examined by an attorney of the purchasing party, a deed for the property was delivered and possession given, and the remainder of the consideration to be paid in three equal annual installments, with 6 per cent interest per annum, to be secured by deed of trust on the property, is a contract for the sale of the property described; and the fact that it was left to the purchaser or his attorney to determine the question of the sufficiency of title did not affect its force as a contract for the sale of the property, for the reason that a purchaser under such circumstances can not arbitrarily or capriciously or in bad faith say there was no title shown when in fact a title was shown; neither does a further provision giving the purchaser the right to have the land surveyed and mapped and to have it examined and reported upon by an engineer before making the first payment affect the validity of the agreement, as the taking of the property by the purchaser was not conditioned upon either a favorable or an unfavorable report of the engineer, and as the evident purpose of this provision was to give the purchaser time in which to make his first payment and also to enable him to ascertain such facts as would help him organize an intended corporation to handle and hold the property; and neither of these provisions, nor bad faith combined, have the effect of making the agreement an option agreement on the part of the purchaser to purchase the land.

. Knisely *v.* Leathe (Missouri), 178 Southwestern, 453, p. 455, June, 1915.

OPTION AGREEMENT TO PURCHASE—ACCEPTANCE.

A contract by which a purchaser undertook and agreed to pay a certain consideration in stipulated payments for mining property on the seller furnishing an abstract of title and delivering the deed, the deferred payments to be secured by a deed of trust, is binding on the purchaser on the performance of the conditions imposed upon the seller, and though it may be regarded as an option agreement to purchase, is accepted by the purchaser where after the delivery of the abstracts and the tender of the deed the purchaser sent his engineer to examine and report upon the mine, and the engineer did examine and make maps of the mine and made full reports to the purchaser.

Knisely *v.* Leathe (Missouri), 178 Southwestern, 453, p. 455, June, 1915.

CONDITIONAL SALE—FORFEITURE—RIGHT TO RECOVER PURCHASE MONEY.

Where a vendor of mineral lands and mining claims declares a forfeiture under the terms of the conveyance, he can not thereafter sue and recover for an unpaid installment of the purchase price.

Croup *v.* Humboldt Quartz & Placer Mining Co. (Washington), 151 Pacific, 493, September, 1915.

SPECIFIC PERFORMANCE OF CONTRACT OF PURCHASE—DESCRIPTION OF CLAIM.

In an action by a seller to compel the performance of a contract to purchase certain mining claims, the complaint is not insufficient either because it fails to describe the mining claims or because it describes them differently from the contract where the contract itself is filed with the complaint as an exhibit, as this sufficiently establishes the identity of the apparently different descriptions.

Ehrhart *v.* Mahony (California), 148 Pacific, 934, May, 1915.

ACTION TO ENFORCE SPECIFIC PERFORMANCE—ALLEGATION AS TO CONSIDERATION.

A vendor of mining claims can not enforce specific performance of a contract of purchase where he fails to allege and prove facts showing that there was an adequate consideration for the obligation sought to be enforced, or that the contract was just and reasonable as to the purchaser, as such facts must be alleged and proved in a suit for the specific performance of a contract of purchase.

Ehrhart *v.* Mahony (California), 148 Pacific, 934, p. 935, May, 1915.

AGREEMENT TO DEVELOP MINE AND RECONVEY—EFFECT OF LACHES.

Where lands supposed to be valuable for minerals were conveyed under a trust arrangement and agreement by which the grantee agreed to develop the lands and operate the same for mining purposes and to receive the issues, rents, and profits, and bound himself to reconvey the lands to the grantor when he had received as such rents and profits and products of the mines the amount of money which he had paid out, together with interest thereon, and where after a long delay there was nothing to show that the mines yielded a profit within a reasonable number of years sufficient to reimburse the grantee, and where subsequently the grantee conveyed an undivided interest in the property to other persons without any declaration of trust, and some 13 years later the land was conveyed by the tenants in common in trust to secure performance of certain covenants entered into by a certain mining corporation, which had agreed to purchase the land for the purpose of mining the same, and subsequently abandoned its rights under the contract, and where subsequent grantees held and occupied the land for a period of more than 30 years, the delay for such time before seeking to enforce the original trust is such laches as would bar relief, and the subsequent conveyance was such a repudiation of the trust as would set in operation the statute of limitations. Neither can the original conveyance of the mineral land on the trust to develop the mine and reconvey when the profits of the mine equaled the original consideration with interest, be regarded as a mortgage where the vendors were not debtors to the vendee, but the transaction partakes more of the nature of a defeasible purchase, but if regarded as a mortgage it would be barred by the statute of limitations.

Coxe *v.* Carson (North Carolina), 85 Southeastern, 224, p. 225, May, 1915.

VENDOR'S LIEN—ACTION FOR PURCHASE PRICE NOT A WAIVER.

Where the vendor of mining property retains the legal title, a proceeding at law for the purchase price is not a waiver of his right to proceed against the property.

Ehrhart *v.* Mahony (California), 148 Pacific, 934, May, 1915.

BROKER'S COMMISSION—RIGHT TO RECOVER.

Where an agent or broker is employed by the owner of mining property to secure a purchaser and the agent brings to the owner a purchaser for such mining property, and the owner enters into a contract of his own making with the purchaser so furnished, the owner thereby accepts the purchaser so found by the broker, and the broker's commission is then due, although it may afterwards turn out that the customer was not financially able to buy.

Knisely *v.* Leathe (Missouri), 178 Southwestern, 453, p. 459, June, 1915.

FRAUDULENT REPRESENTATIONS—CANCELLATION OF INSTRUMENT—ESTOPPEL.

An action to rescind a contract of purchase of a mining lease on the ground of fraud and false representations as to the minerals and quantity of ore can not be maintained where the purchaser became aware of the failure of the mining property in the principal particulars to come up to the representations and where, after acquiring such knowledge, he continued in its ownership and use and thereby elected to stand by the contract, as in order to rescind he must act promptly on first discovering the fraud.

Greenstreet *v.* Walsch (Missouri Appeals), 176 Southwestern, 1062, p. 1063, May, 1915.

FRAUD—BELIEF AS TO EXISTENCE OF CONTRACT OF PURCHASE—RIGHT TO ENFORCE.

A person who is fraudulently led to believe that a certain contract for the purchase of mining property was in writing and contained certain terms and stipulations for his protection and acted upon such belief to his prejudice has the right to enforce the contract against the person defrauding him.

Davenport *v.* Burke (Idaho), 149 Pacific, 511, June, 1915.

SURFACE AND MINERALS—OWNERSHIP AND SEVERANCE.

SEPARATION OF MINERAL AND SURFACE ESTATES.

The owner of mineral lands whether platted or not has the power to convey the surface to one person and the mineral below the surface to another, or he may convey either the surface or the underlying mineral to one person and retain the remaining part of the estate to himself.

Hyde Park Investment Co. *v.* Glenwood Coal Co. (Iowa), 153 Northwestern, 181, p. 184, June, 1915.

BURDEN OF PROOF TO SHOW TITLE IN MINERAL GRANTEE.

Where the description of land conveyed is couched in such general terms that it may cover two or more tracts of land, the ambiguity is a latent one and parol evidence is admissible to show which tract was meant; and the grantee of oil and minerals in and under a part of one tract included in such ambiguous description has the burden of showing, as against a grantee of other parts of such ambiguous description, that his deed covered, and that his mineral rights apply to, the particular tract in controversy.

Virginia Iron, Coal & Coke Co. *v.* Combs (Kentucky), 177 Southwestern, 238, June, 1915.

RESERVATION OF MINERALS IN DEDICATORY PLAT—EFFECT OF RECONVEYANCE BY QUITCLAIM DEED.

The owner of a large tract of land containing valuable coal deposits conveyed the same to an investment company for the purpose of platting it into lots as an addition to the city of Des Moines. At the time of the execution of the deed a mortgage was given by the purchaser to secure the principal part of the unpaid purchase price. The investment company immediately platted the land into lots and in the dedicatory statement accompanying the plat the investment company expressly reserved the coal and minerals of all kinds underlying the entire tract embraced by the plat with the right to mine and remove the coal. Subsequently numerous lots were sold by the company, but the enterprise not proving successful and the mortgagor failing to pay the installments of the mortgage, the original owner, in the course of time through various tax sales and sheriff's sales reacquired title to a large part of the lots composing the tract now in controversy, and the subject matter of the present suit. The original grantee, the investment company, desiring to avoid foreclosure proceedings and a possible deficiency judgment, proposed to and did reconvey by quitclaim deed to the original grantor the part of the tract so reacquired by such tax and sheriff's sales, but no reference in such quitclaim deed was made to the mineral rights or coal, as reserved by the investment company in its original plat. After thus reacquiring title and obtaining the equity of redemption by quitclaim deed from the mortgagor, the original grantee, the original grantor resold the reacquired part to a third person who thereupon leased the same to a coal company for the purpose of developing and mining the coal. The quitclaim deed so executed by the investment company was held sufficient to convey all its rights, title, and interest in and to the land as well as to the mineral rights and coal as against the reservation made by it in its plat of the land, and the investment company had no right or equity on which it could base a suit to enjoin the coal company from mining and remove the coal under its lease from the grantee of the original grantor.

Hyde Park Investment Co. *v.* Glenwood Coal Co. (Iowa), 153 Northwestern, 181, p. 184, June, 1915.

CONVEYANCE OF PLATTED LOTS—EFFECT ON MINERAL RIGHTS AS AGAINST RESERVATION IN PLAT.

The mere platting of the surface of mineral lands in town lots does not affect the nature or solidarity or integrity of the estate of the owner in the land as one individual entity, except as his rights may be affected by the streets and grounds which he has dedicated to the public, but he is still the owner of the property and holds it by precisely the same title as before and may sell and convey the surface above and the mineral below, or separately, as he choses; and if the owner after making and recording such plat conveys a lot by a description such as would be regarded as sufficient to convey the entire title in ordinary cases, there is no conclusive presumption that such conveyance was executed, delivered, and accepted with the understanding that it should convey the surface right only, as against a general reservation of minerals underlying the entire tract expressly stated in the plat.

Hyde Park Investment Co. *v.* Glenwood Coal Co. (Iowa), 153 Northwestern, 181, p. 184, June, 1915.

COAL AND COAL LANDS.

SALE OF COAL IN PLACE—SPACE LEFT BY REMOVAL OF COAL.

Where there has been an absolute sale of the mineral lying under land and a severance of the estate in the mineral from the estate in the surface, the title obtained by the owner of the mineral is an estate in fee, terminating when the mine has been exhausted; and where there are no restrictions in the grant, reservation, or exception which creates the estate, the space which may be left by the removal of the mineral and so much of the containing strata as may be reasonably required for the mining operations, remains a part of the property of the mineral owner only until the exhaustion of the mine and may be used by him during the continuance of the estate as he may see fit, provided that such use does no injury to the surface, and this right of use includes the right to haul mineral and turn water through such space from other mines.

Sharum *v.* Whitehead Coal Mining Co., 223 Federal 282, p. 290, April, 1915.
See Moore *v.* Indian Camp Coal Co., 75 Ohio State, 493, 80, Northeastern, 6.
Lillibridge *v.* Lackawana Coal Co., 143, Pacific 293, 22 Atlantic, 1035.

ACTION FOR POSSESSION—DISCLAIMER—MINERAL RIGHTS.

In an action for the possession of mineral lands without any reference to the severance of the surface and underlying minerals, the plaintiff can not recover as to the specific underlying minerals in the disputed ground, where he makes no claim or demand and offers no proof as to such underlying mineral other than that shown in respect of the soil; and where there was no suggestion in the evidence of a severance between the soil and the minerals, or of a right to minerals more extensive than the soil to which he showed title; and the fact that the defendant disclaimed the mineral rights because they were reserved by his grantor, does not give the plaintiff under his evidence a right to a judgment to the mineral rights separate from his title to the surface as proved by him.

Martin *v.* Howard (Alabama), 68 Southern, 982, p. 983, May, 1915.

CONVERSION—INSTRUCTION AS TO MEASURE OF DAMAGES—DUTY OF PARTY TO REQUEST.

In an action by one land owner against another for mining and converting coal, an instruction to the effect that if the jury believe from the evidence that the defendant mined the coal from the plaintiff's land and converted the coal to its own use and at the time of the mining and conversion the land was the property of the plaintiff, then the plaintiff will be entitled to recover and they should assess his damages at what they think would be reasonable and right under the evidence—not to exceed the amount of his complaint, is not erroneous or prejudical error, though the instruction did not purport to define rules by which the amount should be ascertained under the evidence; and if the defendant conceived that the instruction was deficient in not so defining the method of ascertainment of damages it was its duty to request a definite instruction on the proposition.

Aldrich Mining Co. *v.* Pierce (Alabama), 68 Southern, 901, p. 902, May, 1915.

MINING AND CONVERTING COAL—ADVERSE POSSESSION.

In an action by a land owner against a mining company, the owner of adjoining land, for wrongfully mining and converting coal under the plaintiff's land, the defendant is liable where the evidence shows that the coal was mined under the plaintiff's land, and the defense of adverse possession can not prevail where the evidence shows that the defendant's possession was under claim of right and hostile to plaintiff's possession, only up to the true line between the sections in which the plaintiff's and defendant's lands respectively lay.

Aldrich Mining Co. *v.* Pierce (Alabama), 68 Southern, 901, p. 902, May, 1915.

EJECTMENT—LEGAL TITLE PREVAILS OVER EQUITABLE.

In an action for the possession of coal, iron ore, fire clay, and other minerals on a certain tract of land, proof on the part of the plaintiff showing a prior legal title from a common source will entitle him to recover as against an equitable title from the same common source.

Clinchfield Coal Corporation *v.* Steinman, 223 Federal, 743, p. 746, May, 1915.

ADVERSE POSSESSION INSUFFICIENT.

The masting of hogs or the grazing of cattle on inclosed mineral land will not constitute adverse possession as these are in the same category with other fitful acts of possession, such as the occasional cutting of timber, mowing of hay, or digging of coal, which are generally regarded as insufficient to sustain a plea of limitation.

Kentucky Coal Lands Co. *v.* Wilder (Kentucky), 176 Southwestern, 1155, p. 1156, June, 1915.

OIL AND OIL LANDS—SALE AND CONVEYANCE.

OIL AND GAS AS REAL ESTATE—CONVEYANCE.

For the purpose of ownership and conveyance of solid minerals the earth may be divided horizontally as well as vertically and title to the surface may rest in one person and title to the strata beneath the surface containing such minerals in another; and oil and gas lying within the strata of the earth necessarily are a part of the realty, and being a part of the realty while in place it must legally follow that when they are conveyed while in that condition or possessing that status, a conveyance of an interest in the realty results. There is substantial ground for the distinction sometimes made in this respect between solid minerals and oil and gas, and a purchaser of the oil and gas within the ground assumes the hazard of their absence through the possibility of their escape from beneath a particular tract of the surface and if they are not discovered the conveyance is of no effect; but the possibility of the escape of oil and gas does not render them while in place incapable of conveyance, and their ownership while in that condition with the exclusive right to take them from the land is nothing less than ownership of an interest in the land, and especially so where parties have contracted upon the assumption that they do exist in the land described and their conveyance while in place is consequently a conveyance of an interest in the realty, and their conveyance requires all the formalities of a conveyance of any other interest in the real estate.

Texas Co. *v.* Daugherty (Texas), 176 Southwestern, 717, p. 719, May, 1915.

NATURE OF OIL AND GAS—OWNERSHIP AND POSSESSION.

Oil and gas in the ground, outside of an artificial receptacle, as the casing of a well or pipe line, are parts of the realty underneath the surface where they lie and the owner of the surface is the owner of the oil and gas beneath, but if they stray into the lands of another he ceases to own them, and until they are severed from the realty they are as much a part of it as coal or salt; but the possession of the land is not necessarily the possession of oil and gas for the reason that if an adjoining or even a distant owner drills on his own land and takes oil or gas therefrom, and oil or gas from under the lands of another enters into his well or under his control, it then becomes his property. The owner of the land has only qualified rights to the oil and gas beneath the surface. These include the right to reduce them to possession and to exclude all others exercising the right on his premises, but title in him to such oil and gas does not vest until he has reduced them to actual possession, either by bringing them into a well or into a pipe line, or into a tank or other receptacle in case of oil, as until this is done the gas and oil by natural forces may escape from his land and be reduced to possession as property of another.

Natalie Oil Co. *v.* Louisiana Railway & Navigation Co. (Louisiana), 69 Southern, 146, p. 148, June, 1915.

GRANT OF OIL AND GAS.

A grant of all the oil and gas, coal, and other minerals in and under a particular tract of land described for a stated consideration and certain stipulated royalties, together with the exclusive right of ingress and egress at all times for the purpose of drilling, mining, and operating for such minerals and the erection of appliances, structures, the laying of the necessary pipe lines, and providing that the grantee is "to have and to hold, all and singular, the above-described premises, rights, properties, and privileges, and all such as are hereinafter specified, unto the said grantee, and the heirs, successors, and assigns of such, forever," is not a mere demise of the premises for a given period, nor is it a grant of the right to prospect upon the lands for oil and gas and reduce the same to possession and ownership, but it amounts to a defeasible title in fee to the oil and gas in the ground and the conditions of forfeiture must be taken as conditions subsequent and renders the title subject to be defeated for failure to perform the stipulated conditions.

Texas Co. *v.* Daugherty (Texas), 176 Southwestern, 717, p. 718, May, 1915.

CONTRACT FOR SALE OF OIL LANDS—CONSTRUCTION AND COMPENSATION.

The owner of valuable oil property entered into a contract with another by which the other contracting party was authorized to sell on commission or to buy certain oil property at a stipulated price until a well then in process of drilling should be "drilled in" and "completed," this provision being manifestly intended to protect the owner from depreciation in the event of a "dry hole" or loss from depreciation in the value of his property if the well in process of drilling should prove to be a good well, and such contract and its terms are limited to the time between the date of the contract and the time immediately before the drilling in and completion of such well into and through all the oil and gas bearing sands in the vicinity of the well; and it must be understood that the words "drilled in" and "completed," as applied to the well in process of drilling, are used synonymously and mean one and the same thing, and have reference to the well being drilled in and completed through the oil or gas bearing sands; and the time limit in the contract can not be controlled by any agreement or arrangement between the owner of the well and the drilling contractor, not referred to, nor made with reference thereto, as to what was meant by the words "drilled in" or "completed," as employed in the original contract of agency; and the second party to the contract, in order to entitle him to the stipulated compensation, or to any compensation, must perform the contract and find a purchaser within the time specified.

Chambers *v.* Simmons (West Virginia), 85 Southeastern, 182, April, 1915.

RIGHT OF RAILROAD COMPANY TO DRILL ON RIGHT OF WAY—INJUNCTION BY ADJOINING LANDOWNER.

Where a railroad company is the owner in fee in the land used as its right of way, it has the right to explore the same and drill thereon for the purpose of extracting oil therefrom, and an adjoining landowner is without right to disturb its operation or enjoin it from the exercise of such right; and where, under such circumstances an adjoining landowner seeks to enjoin the drilling operations on the part of the railroad company, a bond is properly required before the injunction issues, so as to compensate the company in damages if it developed later that oil was extracted from beneath the land owned by the railroad company; but the difficulty in the way of proving damages, owing to the peculiar character of the gas and oil which may escape from one place to another, renders such a bond of little use is case of a definitive judgment against the complainant in the suit and an injunction under such circumstances might work an irreparable injury, while on the other hand if the developments continued to the injury of the complainant it would be a small matter to measure the oil and gas extracted through the wells bored on the right of way.

Natalie Oil Co. *v.* Louisiana Railway & Navigation Co. (Louisiana), 69 Southern, 146, p. 148, June, 1915.

See Hayne *v.* Edenborn (Louisiana), 68 Southern, 737, May, 1915.

MINING TERMS.

BATTERY.

A battery is made of three stulls placed together and put in at the pitch of the vein, usually located a few feet apart, up and down, and crosswise of a stope.

Lesh *v.* Tamarack Mining Co. (Michigan), 152 Northwestern, 1021, p. 1022, June, 1915.

MILLS.

Mills are openings in the floor or bottom of a stope through which the ore or mineral is passed or thrown downward along the footwall to the level.

Lesh *v.* Tamarack Mining Co. (Michigan), 152 Northwestern, 1021, p. 1022, June, 1915.

POP SHOTS.

A pop shot is a shot by which a bowlder in a mine is broken up by placing a stick of dynamite on top of the bowlder and exploding it.

Baltesel *v.* American Zinc, Lead & Smelting Co. (Missouri Appeals), 176 Southwestern, 446, p. 447, May, 1915.

PUFFER BOY.

A puffer boy is a person employed to operate an engine in a mine called a puffer engine, used for hauling loaded cars through haulageways.

Lahti *v.* Tamarack Mining Co. (Michigan), 152 Northwestern, 907, June, 1915.

SCRAM.

A scram is a small soft-coal mine complete in itself.

Republic Steel & Iron Co. *v.* Luster (Alabama), 68 Southern, 358, p. 359, April, 1915.

SOLLAR.

A sollar is a place or platform from which trammers shovel or throw the ore or rock into a car.

Lesh *v.* Tamarack Mining Co. (Michigan), 152, Northwestern, 1021, p. 1022, June, 1915.

MINING CORPORATIONS.

INJUNCTION TO PREVENT CONSOLIDATION.

In an action by a minority stockholder of a mining corporation to prevent by injunction a consolidation of a number of mining corporations, the plaintiff makes a prima facie case which the defendants are bound to overcome, when he shows by competent testimony that the consolidation plant and the milling plant proposed and which are being pursued are prejudicial to the rights of the minority stockholders in one company and unduly favorable to the interests of the controlling company seeking to enforce the consolidation.

Hyams *v.* Calumet & Hecla Mining Co., 221 Federal, 529, p. 541 (January, 1915).
Turner *v.* Calumet & Hecla Mining Co. (Michigan), 153 Northwestern, 718, July, 1915.

PROMOTERS OCCUPY FIDUCIARY RELATION TO COMPANY SUBSCRIBERS.

Promoters of a mining corporation formed for the purpose of purchasing from them valuable mining property occupy a fiduciary relation to their cosubscribers and are bound to truthfully declare to their associates any personal interest they may have in the matter of the purchase; and without such disclosures they can not legally profit at the expense of their associates, and the interests of future stockholders are entitled to protection from concealed benefits or profits acquired by the promoters in other transactions with the corporation and to a full disclosure of the true facts of the purchase price of the mining property, and an advantage or benefit accruing to the promoters by failure to do so or concealment constitutes a fraud on the corporation; and the corporation is entitled to set aside the transaction or recover compensation for any loss suffered.

California Calaveras Mining Co. *v.* Walls (California), 149 Pacific, 595, p. 599, June, 1915.

MANDAMUS TO COMPEL DIRECTORS TO CALL MEETING.

The stockholders of a mining corporation may avail themselves of the remedy by mandamus to compel the board of directors to call an annual meeting for the election of directors, and the rule applies to directors of foreign corporations who reside in the State, where the property of the corporation is in the State and where the usual corporate acts have been performed.

Stapler *v.* El Dora Oil Co. (California Appeals), 150 Pacific, 643, p. 644, July, 1915.

CONTROL BY AID OF PROXIES.

The control of a mining corporation purposely gained and exercised by a majority stockholder with the aid of proxies of other stockholders may have the same effect as a control by actual stock majority.

Hyams *v.* Calumet & Hecla Mining Co., 221 Federal, 529, p. 541 (January, 1915).
Turner *v.* Calumet & Hecla Mining Co. (Michigan), 153 Northwestern, 718, July, 1915.

BANKRUPTCY—AUTHORITY OF MANAGING OFFICERS.

An act of bankruptcy on the part of a mining corporation at the instance of the managing director and those acting with him having full control of the company's affairs is sufficient to authorize the appointment of a receiver, though not authorized by a vote of the directors or stockholders.

James Supply & Hardware Co. *v.* Dayton Coal & Iron Co., 223 Federal, 991, p. 994, June, 1915.

LIABILITY ON NOTE EXECUTED BY OFFICERS.

A mining corporation is liable on a note executed by its officers, though the name of the corporation does not appear as a maker, where the evidence shows that the corporation negotiated the loan, received the money, and expended it for its own purposes, and the president and secretary signed the note, intending thereby to execute it as the note of the corporation, and it was accepted as such, and where the execution of the note was subsequently ratified by the directors and stockholders.

San Joaquin Valley Bank *v.* Gate City Oil Co. (California), 149 Pacific, 557, p. 558, June, 1915.

RIGHT OF STOCKHOLDER TO SUE AT LAW WHERE CORPORATION REFUSES.

A stockholder of the United Copper Securities Co. can not, on behalf of himself and all other stockholders of the company, maintain an action to recover treble damages under section 7 of the Sherman Antitrust Act of July 2, 1890 (26 Stat., 40), from certain-named individuals and the Amalgamated Copper Co., on the ground of conspiracy to create a monopoly in the interstate commerce and trade in copper produced in the State of Montana, although he avers that he had requested such United Copper Securities Co. to bring suit and it had refused to do so. There seems to be no ground for holding that a stockholder may bring an action at law in the name of the corporation to recover money damages or specific property where the corporation itself refuses to do so.

United Copper Securities Co. *v.* Amalgamated Copper Co., 223 Federal, 421, April, 1915.

RIGHTS OF MINORITY STOCKHOLDERS—ENJOINING SALE AND CONSOLIDATION.

Minority stockholders of a mining corporation may maintain a suit in equity to set aside a sale of the property of the mining corporation and an attempted consolidation with smaller corporations, where the price paid for the mining property was inadequate, because of the methods of sale, the nature of the consideration and its intended disposition.

Geddes *v.* Anaconda Copper Mining Co., 222 Federal, 129, p. 131, May, 1915.

PURCHASE OF STOCK—CONTROL.

A mining corporation empowered to subscribe for, purchase, own, and dispose of stock in any company organized under the laws of Michigan, for the purpose of mining, refining, smelting, or manufacturing any or all kinds of ores or minerals, has no power to purchase stock of other corporations for the purpose of controlling their management; and the statutory right to stock ownership in another corporation and the control thereby given effect no change in the fiduciary obligation which exists on the part of majority holders toward minority holders in the absence of statutes expressly permitting such ownership.

Hyams *v.* Calumet & Hecla Mining Co., 221 Federal, 529, p. 537 (January, 1915).

Turner *v.* Calumet & Hecla Mining Co. (Michigan), 153 Northwestern, 718, July, 1915.

SALE OF STOCK TO ANOTHER CORPORATION—RIGHT OF STOCKHOLDER.

A stockholder in a mining corporation can insist that any sale of all corporate property upon dissolution shall be to the highest bidder for cash, and not to a corporation in which the majority are interested and for its stock at prices fixed by them.

Geddes *v.* Anaconda Copper Mining Co., 222 Federal, 129, p. 134, May, 1915.

RIGHTS OF MAJORITY AND MINORITY STOCKHOLDERS—EQUITABLE RELIEF.

Independently of State or National antitrust statutes the rule is that a person in control of a majority of the stock and of the board of directors of a mining corporation occupies a fiduciary relation toward the minority stockholders and is charged with the duty of exercising a high degree of good faith, care, and diligence for the protection of such minority interests, and any act in his own interest to the detriment of the minority stockholders is a breach of duty and of trust, and entitles a minority stockholder to plenary relief from a court of equity.

Hyams *v.* Calumet & Hecla Mining Co., 221 Federal, 529, p. 537 (January, 1915).

Turner *v.* Calumet & Hecla Mining Co. (Michigan), 153 Northwestern, 718, July, 1915.

SALE AND DISTRIBUTION OF CORPORATE PROPERTY—RIGHTS OF STOCKHOLDERS.

A stockholder in a mining corporation is not bound to expect anything but money for his equitable share of corporate property, nor is he bound to permit a sale to be made for other chattels or goods to be distributed.

Geddes *v.* Anaconda Copper Mining Co., 222 Federal, 129, p. 134, May, 1915.

SALE OF PROPERTY BY COMMON DIRECTORS—RELIEF IN EQUITY.

The sale of the stock of a mining corporation made by the directors who were also directors in the purchasing company and consented to by a majority of the stockholders, and where large dividends have been subsequently paid on the stock, will not be unconditionally set aside at a suit of the minority stockholders, where the corporation was largely in debt, its mines exhausted, closed, and flooded, and its remaining properties were virgin ground and unworkable zinc-silver ore; but in such case a resale will be ordered if a greater sum can be realized, but if no greater bid than the total proceeds of the sale as originally made, then the original sale may stand.

Geddes *v.* Anaconda Copper Mining Co., 222 Federal, 129, p. 135, May, 1915.

FORECLOSURE OF MORTGAGE—RIGHT OF STOCKHOLDER TO INTERVENE.

In an action by a creditor against a mining corporation to foreclose a mortgage, a stockholder and a third person interested in the premises may, on a showing that he has demanded of the officers of the mining company that they defend the action of foreclosure, but that the officers have neglected to do so and are in a conspiracy with the plaintiff to prevent a defense being interposed and on an averment that the notes and mortgages sued upon were fraudulent and ultra vires, intervene and make such defense on behalf of the mining corporation, and his complaint and petition for intervention may be treated by the court as an answer to the complaint and petition to foreclose.

Investors' Syndicate *v.* North American Coal & Mining Co. (North Dakota), 153 Northwestern, 472, p. 473, July, 1915.

AGREEMENT AS TO ISSUANCE OF STOCK—VIOLATION AND REMEDY.

By an arrangement and agreement three persons, by joint adventure, procured an option to develop a mine, and formed a corporation for that purpose under an agreement by which each should receive one-third of the capital stock of the corporation. The corporation was formed with the capital stock fixed at 500,000 shares of the par value of $1 each and $250,000 was made treasury stock, and thereupon one of the parties caused to be issued to himself 125,000 shares and to each of the other two members 62,500 shares. Under the original agreement and without making any treasury stock each of the ventures was entitled to receive 83,333⅓ shares; but after deducting the treasury stock the issuance of the 125,000 shares to one of the parties was a violation of the agreement, and the others had the right to refuse the 62,500 and to compel the party receiving the 125,000 shares to transfer 20,833⅓ shares; but it was an error for the trial court to award one of them 41,666⅔ shares of the 125,000 so issued to one of the parties.

Kent *v.* Costin (Minnesota), 153 Northwestern, 874, p. 875, July, 1915.

AUTHORITY OF AGENT—FAILURE TO COMMUNICATE INSTRUCTIONS.

A general superintendent of a milling company instructed and directed to employ a person to do certain hauling and to have certain machinery installed has power to bind the company to the terms of the contract made by him for the purpose of accomplishing the work, and the party with whom he contracts is not bound by the limitations placed upon the agent as to the price to be paid for the work, where such instructions as to price were not communicated to him.

Bonanza Milling Co. *v.* Borego (Colorado), 148 Pacific, 859, p. 860, May, 1915.

SERVICE OF PROCESS—DOING BUSINESS WITHIN A STATE.

The statute of Washington (Remington and Ballinger's Code, section 225, subdivision 9), providing that summons may be served on a foreign corporation doing business within the State, does not apply to a corporation organized in the State of Minnesota and conducting mining operations in Alaska, and is maintaining in Seattle, in the State of Washington, a purchasing and forwarding agent on a salary, the corporation paying the office rent and expenses, and where such agent has purchased goods, wares, and merchandise in the city of Seattle with directions that they be shipped to the defendant corporation at its place of business in Alaska, and where such agent is charged also with the duty of seeing that the goods ordered by the defendant corporation from eastern points or cities are transshipped at Seattle and forwarded to the place of business of the corporation in Alaska, and where such agent does not pay for any goods or disburse any moneys whatsoever, but where moneys are disbursed from the general office of the defendant corporation at places outside of the State of Washington, as in such case the corporation is not doing business within a State.

Johnson *v.* Alaska Treadwell Gold Mining Co., 225 Federal, 270, April, 1915.
See Daly *v.* Lahontan Mines Co. (Nevada), 151 Pacific, 514, September, 1915.

MANDAMUS TO CONTROL NATURAL GAS COMPANY.

A corporation organized prior to the passage of the act approved March 26, 1913 (Session Laws of Oklahoma, 1913, ch. 99), as a gas purchasing, producing, transporting, and delivering company, and at the time of the passage of the act referred to the corporation had acquired gas-producing wells throughout the State and was engaged in the business of transporting natural gas from such wells through a system of pipe lines to the limits of certain cities in the State and there selling and delivering its natural gas to domestic corporations with which it was in nowise connected. The wells of the corporation in its several gas fields all drew from the same source of supply as other wells in the same field owned and operated by other producers, and so much as any one well produced the supply was diminished, but the gas when reduced to possession could not be profitably stored. While the corporation was so operated the act of March 26, 1913, was passed making it unlawful for the corporation to engage in the business of purchasing, producing, transporting, and delivering natural gas within the limits of the State, except as authorized by and subject to the provisions and restrictions of such act; and the act permitted the corporation to continue its business as a common carrier and a common purchaser by first accepting the provisions of the act in a certain manner within a certain time, but on the failure of the corporation to do so, mandamus will not lie to compel it to accept the provisions of the act and operate thereunder as a common carrier and common purchaser, nor will mandamus lie to compel a general course of conduct and a long series of continuous acts, where it is not possible for the court to oversee the performance of the company's duties as prescribed in the act, and where the act affords the State a plain, adequate, and complete statutory remedy against the corporation.

Oklahoma Natural Gas Co. *v.* State (Oklahoma, 150 Pacific, 475, June, 1915.

ACTS OF BANKRUPTCY—INSOLVENT CORPORATION AS DEFENDANT.

Where a receivership was procured by actual authority of a mining corporation and on its behalf, as a defendant in a pending suit, it was as effectual an act of bankruptcy as if the suit had been directly in the name of such corporation as complainant; and it is immaterial that the receivership was not ordered because of insolvency if the mining corporation showed actual insolvency at the time the receivership was applied for.

James Supply & Hardware Co. *v.* Dayton Coal & Iron Co., 223 Federal, 991, p. 993, June, 1915.

SUFFICIENCY OF APPLICATION FOR RECEIVER.

A petition by certain minority stockholders of an oil company averring that the corporation owned property of the value of $100,000 and owed debts in the sum of about $15,000, and that among that corporation's assets was a claim or cause of action against certain named persons in the sum of about $20,000, which claim was for stock-promotion expenses and commissions and cash wrongfully and fraudulently obtained by such named persons, as fully shown in a prior report of an auditor, and that such named persons were disposing of their stock in the corporation for the purpose either of controlling the corporation and having such claims canceled or to prevent the corporation from bringing suit to collect such claims, and also averring that if suit was not brought the claims would be barred by the statute of limitations, and averring also that the managing officers of the corporation declined and refused to bring suit, is not sufficient to authorize the appointment of a receiver for the corporation where no cause of action is asserted in the petition against any of the defendants, nor any recovery sought in the suit, nor any relief prayed for except the appointment of a receiver, as the minority stockholders themselves could, on a proper showing, bring suit to recover for the benefit of the corporation.

Forest Oil Co. *v.* Wilson (Texas Civil Appeals), 178 Southwestern, 626, p. 628, June, 1915.

APPOINTMENT OF RECEIVER—PRIMA FACIE CASE.

Section 8, chapter 51, of the laws of 1909 of South Dakota, authorizing the appointment of a receiver in a suit commenced for the foreclosure of a miner's lien upon mining property and making it the duty of a court in such case to appoint a receiver does not mean that simply upon the filing of such suit and a demand for the appointment of a receiver the court is authorized to make such appointment, as the effect of the appointment of a receiver is to take from the owner the right of possession and control of his property and place it under the control of a stranger, and any statute permitting or authorizing such act without a showing justifying it is fundamentally and clearly void; but the provisions of this act, so far as they relate to the appointment of receivers, should be construed in the light of other existing laws in relation to receivers, and before a court would be authorized to appoint a receiver in such an action a showing upon notice should be made sufficient to justify and warrant the appointment.

Cessna *v.* Otho Development & Power Co. (South Dakota), 153 Northwestern. 380, p. 382, June, 1915.

RECEIVER—POWER OF COURT TO DECREE PRIORITY OF LIEN.

A court of equity appointing a receiver of a mining corporation has no power in the absence of the consent of prior contract lien creditors, in authorizing its receiver to continue the business of the company, to decree that the indebtedness of a particular defendant shall take precedence over such prior contract liens as a mortgage in the absence of the consent of the mortgagee.

Stacey *v.* McNicholas (Oregon), 148 Pacific, 67, p. 71, April, 1915.

RECEIVER'S SALARY AND EXPENSES AS FIRST LIEN.

Property of a mining corporation properly placed in the hands of a receiver by order of a court of equity becomes chargeable with the necessary expenses incurred in taking care of and saving it, and such expenses, together with the compensation for the services of the receiver, are taxable as part of the costs and entitled to priority of payment as a first lien upon the mining property.

Stacey *v.* McNicholas (Oregon), 148 Pacific, 67, p. 71, April, 1915.

RECEIVER—FUND FOR PAYMENT OF OPERATING EXPENSES.

Where a receiver is appointed by a court for a mining corporation and is by the order directed to continue its business, the claims for expenses in operating the mine not shown to be necessary for the care and preservation of the property should be paid as far as possible from the income realized from the mining operations.

Stacey *v.* McNicholas (Oregon), 148 Pacific, 67, p. 71, April, 1915.

CONTRACT FOR SALE OF MINING STOCK—SILENCE NOT A RATIFICATION.

In an action to recover the balance due on a contract for the sale of certain mining stock in the absence of evidence to the effect that the agent, a brother of the seller, agreed that a certain sum of money used to develop the mine should be applied on the contract, the mere silence of the seller can not be taken as a ratification of a contract that he claimed did not exist.

Clough *v.* Monro (Washington), 150 Pacific, 1190, p. 1192, August, 1915.

EMINENT DOMAIN.

MINING A PUBLIC USE.

By section 2456 of the Revised Statutes of Nevada mining for gold, silver, copper, lead, cinnabar, and other valuable minerals is recognized as the paramount interest of the State and is declared to be a public use.

Goldfield Consolidated Milling, etc., Co. *v.* Old Sandstrom Annex Gold Mining Co. (Nevada), 150 Pacific, 313, p. 316, July, 1915.

APPROPRIATION OF LAND FOR MINING PURPOSES—CONSTRUCTION AND VALIDITY OF STATUTE.

The validity of statutes for the appropriation of private property for mining purposes may sometimes depend upon many different facts, the existence of which would make a public use, even by an individual, where in the absence of such facts the use would be clearly private; but such facts must be general, notorious, and acknowledged in the State, and are not the subject of judicial investigation, but are well known by the courts; and the courts in the construction of such statutes will notice the situation and conditions leading to the demand for the enactment of such statutes, and in their construction may consider the results upon the growth and prosperity of a State which in all probability would flow from a denial of the validity of such statutes, and all such matters may have a material bearing upon the question whether the individual use proposed is not in a fact a public use. But for the existence and validity of such statutes, the owners of mines and of works for the reduction of ores, the operations of which furnish thousands of men in the State with employment at good wages, and to which the general prosperity of the State is largely due, would be denied the right to invoke such statutes when necessary to the successful operation of their business, or for acquiring rights of way for the transportation of ores from the mines to the mills and smelters, or for the construction of tunnels for drains, or for necessary lands for the deposit of tailings; and parties holding title to ground necessary and suitable for these purposes, which might be entirely worthless except for such purposes, would be clothed with power to demand and compel payment of unconscionable prices for their lands before parting with the title, or they could refuse absolutely to grant the easement required on any terms, and thereby cripple mining enterprises or destroy them altogether. Courts may know that such a policy would not only be inconsistent and unreasonable, but would greatly retard the development of one of the greatest natural resources of the State; and in consideration of such facts, persons and corporations owning and operating mines and mills for the reduction of ores may, under such statutes, condemn land for obtaining water for mining purposes, the construction of rights of way for tunnels, flumes, and dumping places for tailings, where the statute makes ample provision for the payment of a fair price to the owner for lands sought to be condemned, as well as for all damages that he may suffer because of the appropriation and use.

Goldfield Consolidated Milling, etc., Co. *v.* Old Sandstrom Annex Gold Mining Co. (Nevada), 150 Pacific, 313, p. 316, July, 1915.

Clark *v.* Nash, 198 U. S., 361, 25 S. Ct., 676.

Strickley *v.* Highland Boy Gold Mining Co., 200 U. S., 527, 26 S. Ct., 301.

Highland Boy Gold Mining Co. *v.* Strickley, 28 Utah, 215; 78 Pacific, 296; 107 Am. St., 711; 1 L. R. A. (N. S.), 976.

NECESSITY OF CONDEMNATION.

In a proceeding by a corporation operating a mill for the reduction of ores to condemn lands for the deposit of its tailings, the law does not require that an absolute necessity should exist for the identical lands sought to be condemned, but it is sufficient if the lands sought to be used will be of great benefit and advantage to the mining industry of the particular community; that it is necessary to condemn such lands for the protection and advancement of these interests, and that the benefits arising therefrom are of paramount importance as compared with the individual loss or inconvenience to the owner of the land; and generally under such circumstances the discretion of the corporation in the selection of land for its use will not be questioned where it acts in good faith and not capriciously.

Goldfield Consolidated Milling, etc., Co. *v.* Old Sandstrom Annex Gold Mining Co. (Nevada), 150 Pacific, 313, p. 318, July, 1915.

INTEREST IN LAND APPROPRIATED.

The statute of Nevada provides for the right of eminent domain for certain public uses and provides also for the appropriation of the fee of land, but the statute does not say that the fee simple shall be taken, but only that it is subject to be taken, and in condemnation proceedings only such an interest as is necessary can be taken.

Goldfield Consolidated Milling, etc., Co. *v.* Old Sandstrom Annex Gold Mining Co. (Nevada), 150 Pacific, 313, p. 319, July, 1915.

APPROPRIATION OF LAND FOR DEPOSIT OF TAILINGS.

The statute of Nevada, section 5606, Revised Statutes, provides that the right of eminent domain may be exercised in behalf of such public uses as tunnels, ditches, flumes, pipes, and dumping places to facilitate the milling and smelting or reduction of ores or the working of mines and for all mining purposes as well as for outlets, natural or otherwise, for the deposit or conduct of tailings, refuse, or water from mills, smelters, or other works for the production of ores, or from mines, milldams, natural-gas or oil pipe lines, tanks, or reservoirs (statutes of 1907, p. 279), and under this section, together with sections 2456 and 2458, a corporation engaged in mining and milling and reducing by other methods gold, silver, and other ores has the right to condemn land for the deposit of the tailings from its mills.

Goldfield Consolidated Milling, etc., Co. *v.* Old Sandstrom Annex Gold Mining Co. (Nevada), 150 Pacific, 313, p. 316, July, 1915.

DEPOSIT OF TAILINGS WITHOUT LANDOWNER'S CONSENT—RIGHT OF SUBSEQUENT APPROPRIATION.

A mining corporation engaged in milling and the reduction of ores, depositing the tailings from the treatment of its ores upon a portion of its own land lying in a gulch and there conserved the same for the purpose of retreatment, but where by reason of high waters and floods large parts of such tailings were washed down upon the land of an adjoining owner, has the right under the statute of Nevada (Revised Laws, sections 2456, 2458, and 5606) to condemn the land or a sufficient part thereof for a right of way for a tramway for the purpose of erecting a tram thereon to reconvey such tailings to its mill; and the common-law rule to the effect that a structure erected by a tort feasor upon the lands of another becomes a part of the land does not apply.

Goldfield Consolidated Milling, etc., Co. *v.* Old Sandstrom Annex Gold Mining Co. (Nevada), 150 Pacific, 313, p. 317, July, 1915.

APPROPRIATION OF SURFACE OF MINING CLAIM.

The fact that a tract of land sought to be appropriated for deposit of tailings by a mining corporation operating a mill and reduction works is a patented mining claim will not defeat the proceedings for appropriation where it appears that the claim was not in fact being worked and had not been worked for several years; and the mere possibility that the land may some time in the future be used by the owner for mining purposes will not prevent condemnation of a right of way for a tramway, and especially where the use for which the condemnation is sought will not interfere with the operation of the land as a mining claim.

Goldfield Consolidated Milling, etc., Co. *v.* Old Sandstrom Annex Gold Mining Co. (Nevada), 150 Pacific, 313, p. 319, July, 1915.

MINING CLAIMS.

NATURE AND GENERAL FEATURES.

NATURE OF STATE STATUTES.

The disposition of mining ground is wholly within the control of the Federal Government, and State statutes for regulation of the location of mining claims and for protection of the possession thereof are statutes of peace and repose, intending to prevent disorder in claiming and holding mining ground.

Florence-Rae Copper Co. *v.* Kimbel (Washington), 147 Pacific, 881, p. 884, January, 1915.

METHOD OF ACQUIRING CLAIM.

Congress has provided how mining claims can be acquired, and this may be done by discovery of mineral, gold, silver, or copper, and the like, upon the public lands and by staking the same off or marking it upon the ground.

Trinity Gold Dredging & Hydraulic Co. *v.* Beaudry, 223 Federal, 739, p. 741, May, 1915.

SIGNIFICANCE AND RELATION.

A mining claim in its accepted significance relates to that portion of the public mineral land which the miner takes up and holds in accordance with the mining law and local statutes and regulations, for mining purposes.

Trinity Gold Dredging & Hydraulic Co. *v.* Beaudry, 223 Federal, 739, p. 741, May, 1915.
See United States Mining Statutes Annotated, 179.

MINING CLAIM AS PROPERTY.

A mining claim when perfected is declared to be property in the highest sense of the term, and may be bought, sold, and conveyed, and will pass by descent.

Trinity Gold Dredging & Hydraulic Co. *v.* Beaudry, 223 Federal, 739, p. 741, May, 1915.
See United States Mining Statutes Annotated, 32, 98, 122, 188, 701.

Mining claims are property in the fullest sense of the word, distinct from the land itself, vendable, inheritable, and taxable.

Earhart *v.* Powers (Arizona), 148 Pacific, 286, p. 287, May, 1915.

LOCATION OF MINING CLAIM.

ORIGINAL OR RELOCATION—QUESTION OF FACT.

Where a mining claim is relocated as abandoned or forfeited ground, such relocation admits the validity of the prior location, and the issue is whether the prior locator has lost his right by forfeiture or by abandonment; but where a subsequent locator bases his right upon the contention that the prior locator never made a valid location under the law, then he is not relocating a forfeited or abandoned claim, but is making an original location of a claim, the prior attempt at which was invalid. In such case the issue is not whether the prior locator has lost a possessory right once legally established, but whether the prior locator ever established a legal right; and the Arizona statutes of 1901, paragraph 3241, has no application, and it not only would not be proper for the new locator to state in his location notice that he located the claim as abandoned property, but such statement if made would preclude him from contesting the question as to the validity of such prior location, the very fact or point he denies.

Copper Queen Consolidated Mining Co. *v.* Stratton (Arizona), 149 Pacific, 389, p. 392, June, 1915.
See Cunningham *v.* Pirrung, 9 Arizona, 288, p. 293; 80 Pacific, 330.

LOCATION OF ABANDONED CLAIMS.

When mineral ground has once been segregated from the public domain by a valid mining location, and thereafter reverts to the public domain by reason of the abandonment or forfeiture of the location by the first locator, a subsequent locator of such abandoned or forfeited ground so opened to location acquires no rights thereto unless he located such ground as abandoned property and such ground can not be otherwise located by any one.

Copper Queen Consolidated Mining Co. *v.* Stratton (Arizona), 149 Pacific, 389, p. 391, June, 1915.

OVERLAPPING LOCATION—EFFECT AND VALIDITY.

The acts of a second locator in locating his claims, so far as they overlap or conflict with existing claims, are ineffectual for the purpose of vesting any right thereto in such locator unless there had been an abandonment of such existing claims or a forfeiture of the rights of the first locator by reason of the failure to do the annual assessment work for the year.

Musser *v.* Fitting (California Appeals), 148 Pacific, 536, p. 537, March, 1915.

VALID LOCATION AS PRIMA FACIE TITLE.

The performance of the annual labor on a mining claim is not necessary except to protect the rights of the locator against parties seeking to initiate title to the same premises; and as against an attempted subsequent location the original locator makes a prima facie case by showing a valid location.

Lancaster *v.* Coale (Colorado Appeals), 150 Pacific, 821, July, 1915.
See United States Mining Statutes, Annotated, 256.

LOCATION NOTICE AND CERTIFICATE.

LOCATION NOTICE—CONTENTS.

The requirements as to the contents of the location notice of a mining claim as provided for by section 7359 of the statutes of Washington (Remington and Ballinger Code), applies only to an original or new location and does not apply as to the contents of a relocation notice under section 7365.

Florence-Rae Copper Co. *v.* Kimbel (Washington), 147 Pacific, 881, p. 884, January, 1915.

OBJECTS AND SUFFICIENCY.

The objects and functions of a location notice do not extend to conferring full title to mining properties, and such a notice differs from the ordinary documentary muniments of title in that it is not a title nor proof of title, nor does it constitute or of itself establish the possessory right to which it relates. It is purely a creature of the statute and its purposes and functions are twofold: 1. When duly recorded it becomes notice to the world of the facts therein set forth and is constructive notice of the claimant's possession. 2. It is by statute made one of the steps requisite to constitute a perfected mining location.

Copper Queen Consolidated Mining Co. *v.* Stratton (Arizona), 149 Pacific, 389, p. 393, June, 1915.

ORIGINAL OR RELOCATION—ABANDONED PROPERTY.

Before its amendment paragraph 3241 of the Revised Statutes of Arizona, 1901, provided that if a location was made on abandoned property the location notice should state such fact, but the statute never required the location notice of an original location to so state, nor did it require the locator of open, unappropriated, public mineral land to so state in his location notice when he located the same as an original location and not as abandoned or forfeited property, though the ground was actually abandoned and forfeited at the time of the controverted location.

Copper Queen Consolidated Mining Co. *v.* Stratton (Arizona), 149 Pacific, 389, p. 392, June, 1915.

POSTING AND RECORDING—PURPOSE AND TITLE.

The object of posting and recording mining notices is to protect a locator and give him an opportunity to develop the property, obtain the ore, and eventually to get title to the land where he continues the improvements to a sufficient extent; and a locator must rely upon his own right or title and can not strengthen the same by tacking on stale claims of other persons.

Richen *v.* Davis (Oregon), 148 Pacific, 1130, p. 1132, May, 1915.

POSTING AND RECORDING AS STEPS TO LOCATION.

A location notice or certificate containing all the matters provided for by paragraph 3232 of the Revised Statutes of Arizona, 1901, and recorded within 90 days after the commencement of the location as required by paragraph 3234 of these statutes, is constructive notice of the location; and when posted on the claim as required, together with the recording, it is one of the steps requisite to constitute a perfected mining location and thereby performs the functions for which the law intended it to serve.

Copper Queen Consolidated Mining Co. *v.* Stratton (Arizona), 149 Pacific, 389, p. 393, June, 1915.

AMENDMENT.

A location notice if defective when posted and recorded, because it fails to contain the statutory recitals required by section 3241 of the Revised Statutes of Arizona, 1901, is not void but voidable only, and is subject to amendment under section 3238 of the statute. This right of amendment is based on the fact that a full, complete, and unimpeachable certificate can not be made without the aid of a surveyor, and persons familiar with the making of mining locations know that usually the first record is imperfect, and recognizing these difficulties it is not the policy of the law to avoid a location for defects in the notice, but rather to give the locator an opportunity to correct his records whenever defects are discovered.

Copper Queen Consolidated Mining Co. *v.* Stratton (Arizona), 149 Pacific, 389, p. 393, June, 1915.

DISCOVERY.

LOCATION WITHOUT DISCOVERY—EFFECT AND VALUE.

A mining location under the United States statutes, without discovery of minerals, can not be said to be totally invalid and of no effect, as the title by such location and possession is good as against every person contending against it, except the Government of the United States; and a transfer of such a location gives the transferee the right to proceed to prosecute work with a view of making a discovery of oil and such possession can not be disturbed by strangers, and is good and the right to such possession is sufficient as a consideration for a lease.

Hullinger *v.* Big Sespe Oil Co. (California), 151 Pacific, 369, p. 370, August, 1915.

APEX OF VEIN.

DEFINITION.

An apex of a vein is that part or portion of the terminal edge of a vein from which the vein has extension downward in the direction of the dip and the definition involves the elements of terminal edge and downward course therefrom; but the locality of the terminal edge is a question of fact and the downward course of a vein has no significance whatever independently of the terminal edge of the vein.

Stuart Mining Co. *v.* Ontario Mining Co., 237 U. S. 350, p. 360, April, 1915.

EXTRALATERAL RIGHTS.

STRIKE AND DIP OF VEIN.

The United States mining statutes, section 2322, calls for no effort of construction, and the distinction which obtains in the parlance of miners and in the cases between the strike or course and the dip of a vein, is compelled by the statute and marks accurately the linear and extralateral rights of a location and the language of the section expresses the distinction which can be observed, and the strike and the dip of the vein must not be confounded nor the rights dependent upon them confused.

Stuart Mining Co. *v.* Ontario Mining Co., 237 U. S., 350, p. 353, April, 1915.

RIGHT TO FOLLOW VEIN THROUGH SIDE LINES.

The locator of a mining claim under section 2322 of the United States Revised Statutes has the right to the surface within the lines of his claim, and if a vein has its top or apex within the claim he may follow such vein downward, though it depart from a perpendicular in its downward course outside the vertical side lines of his location in the adjoining ground.

Stuart Mining Co. *v.* Ontario Mining Co., 237 U. S., 350, p. 352, April, 1915.
See United States Mining Statutes Annotated, 133.

VEIN APEXING WITHIN LOCATION—QUESTION OF FACT.

The locator or owner of a mining claim in seeking to recover the value of ore bodies taken and appropriated by another on a vein outside the vertical planes of the complaining locator must show that the vein from which the ores was alleged to have been taken had its apex within the surface boundaries of the lines of his location and that his is the senior location; but where the court or jury trying the case found as a fact that the particular vein from which the ore was taken as complained of did not apex in the claim of the complaining locator, a court on appeal can not say as a matter of law that the complaining locator is entitled to recover such ore bodies, as in such case the essential fact for the purpose of his recovery is determined against him.

Stuart Mining Co. *v.* Ontario Mining Co., 237 U. S., 350, p. 358, April, 1915.

RIGHT TO FOLLOW VEIN—LIABILITY FOR TRESPASS—AGREEMENT AS TO LOCATION.

In an action by the owner of a mining claim against the owner of an adjoining claim to recover the value of ores alleged to have been taken by the defendant from veins apexing in the plaintiff's claim and extending on their dip outside of the vertical side lines of his claim the plaintiff alleged that he was in possession and entitled to the possession of such named veins throughout their entire depth, but that the defendant, without right, entered upon such veins, ousted the plaintiff therefrom, and has ever since held and continues to hold possession thereof and has extracted therefrom ores of a stated value. In such action a stipulation and an agreement by which the issue of title and ownership and right of possession of the veins and ores in controversy shall be alone tried and determined, and the issue of damages shall be reserved for further trial and proceedings, must be held to relate only to the title and right to possession of the ore in the particular veins mentioned by name in the plaintiff's complaint and petition and put in issue by the pleading; and in a subsequent trial for damages the defendant is entitled to prove, as against the claim for damages, that a large portion of the ore in controversy which was taken from his claim was from other veins that did not apex in plaintiff's claim and for which he could not be required to account to the plaintiff, and the defendant was not estopped by the trial and judgment in the former case as to the title from litigating the question of the amount and value of the ores extracted from the veins named in the plaintiff's complaint and petition.

Work Mining & Milling Co. *v.* Doctor Jack Pot Mining Co., 222 Federal, 216, February, 1915.

OWNERSHIP OF ORES AT INTERSECTION OF VEINS.

The owner of a senior location owns all the ore in a vein apexing within his location and owns all the ore at the point of intersection of his vein and a vein apexing in the junior location and is not subject to the charge of being a trespasser while extracting and removing the ore at such point of intersection.

Esselstyn *v.* United States Gold Corporation (Colorado), 149 Pacific, 93, p. 95, June, 1915.

ASSESSMENT WORK.

EXPENSES INCURRED IN MOVING MACHINERY.

The expenses of getting heavy machinery to a mining claim and a mine thereon which, when in use, will tend to the development of the claim will be allowed on the annual assessment work on such claim, although the machinery and expenses incurred are not within the boundaries of the claim.

Florence-Rae Copper Co. *v.* Kimbel (Washington), 147 Pacific, 881, p. 885, January, 1915.

WORK DONE BY STOCKHOLDERS FOR CORPORATION.

Work done by stockholders for a mining corporation and employees engaged by them for a corporation owning mining claims is a sufficient performance of assessment work, as the stockholder has such an equitable and beneficial interest in the property by reason of which the assessment work done by him inures to the benefit of the corporation and prevents a forfeiture.

Florence-Rae Copper Co. *v.* Kimbel (Washington), 147 Pacific, 881, p. 885, January, 1915.
Musser *v.* Fitting (California Appeals), 148 Pacific, 536, p. 537, March, 1915.

RESUMPTION AND PERFORMANCE—NATURE OF IMPROVEMENTS.

The penalty for failure to comply with the requirements of the United States statutes in respect to the performance of annual labor is that the location shall be opened to relocation in the same manner as if no location had ever been made; but if the original locator has resumed work and is in good faith engaged in the performance of the assessment work, the claim is not subject to relocation. It is sufficient in the performance of such work and the making of improvements that engines, wire cables, and labor used in moving and installing the same were intended primarily for the development and operation of the claim, though they were to be secondarily used for the purpose of aiding and constructing a railway that was itself intended to be an aid and cooperator with the mining claims.

Florence-Rae Copper Co. *v.* Kimbel (Washington), 147 Pacific, 881, p. 884, January, 1915.

RESUMPTION WHEN RELOCATOR FAILS TO PERFORM ASSESSMENT WORK.

The failure of a locator of a mining claim to perform the assessment work required by statute within the year renders the claim subject to relocation; but the fact that another subsequently enters upon and attempts to relocate the claim and himself fails to perform the assessment work for the succeeding year does not prevent the original locator from resuming work on the claim and thereby saving the claim from further relocation, as it is against intervening rights only that the original locator can resume work and resuscitate his possessory rights.

Richen *v.* Davis (Oregon), 148 Pacific, 1130, p. 1132, May, 1915.

RESUMPTION PREVENTS RELOCATION.

The fact that the locator of a placer-mining claim cleared and burned the brush and débris thereon for the purpose of dredging the claim may be taken as a fair indication of the good faith of the locator in maintaining the claim, and together with the money expended in the same direction negatives any intent to abandon the mining claim, and where it shows a resumption of the required labor it will defeat an attempted subsequent relocation.

Richen *v.* Davis (Oregon), 148 Pacific, 1130, p. 1133, May, 1915.

NATURE AND PROOF OF PERFORMANCE.

The more profitable and practical way of extracting mineral from placer mining claim is by means of a dredge, and in order to do so it is absolutely necessary to clear off the brush and timber; and where the surface of a placer claim was covered with a thick growth of brush, with some larger timber, considerable débris which had been deposited by a river during high water, and the labor performed in clearing the surface of such brush, timber, and débris may properly be included as annual assessment work on the mining claim that will prevent a forfeiture and relocation of the claim.

Richen *v.* Davis (Oregon), 148 Pacific, 1130, p. 1131, May, 1915.

STATUTORY AFFIDAVIT AS PRIMA FACIE PROOF.

The affidavit provided by section 1426m of the Civil Code of California constitutes prima facie evidence of the performance of the annual assessment work upon a mining claim; and if such prima facie case is not overcome by proof, then the fact of the performance of the assessment work must be taken as established.

Musser *v.* Fitting (California Appeals), 148 Pacific, 536, p. 536, March, 1915.

FORFEITURE.

WHAT CONSTITUTES AND WHAT PREVENTS.

The term "forfeiture" does not appear in the United States statutes providing for the relocation of mining claims; but the courts employ the term as a comprehensive word indicating a legal result flowing from a breach of condition subsequent, subject to which the locator acquires his title; but courts do not incline to the enforcement of this class of penalties, and a forfeiture of a mining claim does not ensue from the mere failure to comply with the law, but it requires the intervention of a third person and a relocation of the ground before any forfeiture can arise, and when thereby such forfeiture becomes effectual the estate of the original locator is hopelessly lost, and there is no possibility of its being restored. The reason of this rule is that the resumption of the annual assessment work at any time prior to the lawful inception of an intervening right prevents a forfeiture.

Florence-Rae Copper Co. *v.* Kimbel (Washington), 147 Pacific, 881, p. 885, January, 1915.

DEGREE AND BURDEN OF PROOF.

A person seeking to avail himself of the failure of a preceding locator to comply with the law in order to secure a relocation of a mining claim must establish such failure by clear and convincing proof, and a court will construe a mining regulation or custom so as to defeat a forfeiture, if it can, and every reasonable doubt will be resolved in favor of the validity of a mining claim as against the assertion of a forfeiture. The burden of establishing a forfeiture rests upon the party charging it or claiming benefits by reason of an alleged forfeiture.

Florence-Rae Copper Co. *v.* Kimbel (Washington), 147 Pacific, 881, p. 885, January, 1915.
Musser *v.* Fitting (California Appeals), 148 Pacific, 536, p. 537, March, 1915.
Richen *v.* Davis (Oregon), 148 Pacific, 1130, p. 1131, May, 1915.

FAILURE TO PERFORM ASSESSMENT WORK—PROOF.

In order to establish the forfeiture of a mining claim it must be shown by clear and convincing proof that the former locator or owner failed to have the work performed or the improvements made as required by statute, as forfeitures are not favored in such cases.

Richen *v.* Davis (Oregon), 148 Pacific, 1130, p. 1131, May, 1915.

ASSESSMENT WORK PERFORMED BY COOWNER—FORFEITURE.

The only labor or improvement required by section 2324 of the United States Revised Statutes which will entitle a coowner doing the work or making the improvement to forfeit a delinquent coowner's interest is the expenditure of the full sum required by the statute, and this is not less than $100 for each claim. The expenditure required by the statute for 175 mining claims for one year would be $17,500 and for two years $35,000; and an expenditure of $5,600 by one coowner upon the 175 mining claims will not authorize such coowner to forfeit the interest of a coowner that fails to contribute.

Pack *v.* Thompson, 223 Federal, 635, p. 637, May, 1915.

SUFFICIENCY OF NOTICE TO COOWNER.

A notice by coowners professing to perform assessment work on mining claims held in common to another coowner is not sufficient to forfeit the interest of the noncontributing coowner where it conclusively appears from the notice that only a small portion of the assessment work required by the statute had been performed, for the reason that the notice shows that all the assessment work had not been performed and the mining claims were then subject to relocation, and no interest was saved by a partial compliance with the statute.

Pack *v.* Thompson, 223 Federal, 635, p. 638, May, 1915.

FORFEITURE PROCEEDINGS ENJOINED.

Where several coowners of 175 mining claims owned in common served notice on one coowner for the purpose of forfeiting his interest for failure to contribute where the amount stated was less than the required statutory amount for all the claims, and where the same coowners served another notice on such other coowner for failure to contribute his part of the assessment work of $1,200 expended by them on 12 claims, the same being a part of the $5,600 mentioned in the other notice, and the 12 claims being a part of the 175 claims described in the other notice, the notices are so inconsistent, defective, and insufficient, and one notice so discredits the other that it will justify a court of equity in suspending by temporary injunction the forfeiture proceedings until the actual facts can be ascertained and the questions involved determined upon the merits.

Pack *v.* Thompson, 223 Federal, 641, May, 1915.

ABANDONMENT.

INTENT.

Abandonment of a mining claim is a question of intent.

Richen *v.* Davis (Oregon), 148 Pacific, 1130, p. 1132, May, 1915.

WHAT CONSTITUTES—KNOWLEDGE OF RELOCATOR.

Where it appears that several thousand dollars had been expended in furnishing and moving engines and other material and in building and improving trails and roads for the better development of a mining claim and the mine thereon, commencing as early as May and continuing without interruption until July of the year following, this is sufficient not only to show a performance of the assessment work but also to show that the locator had no intention of abandoning the mining claim; and a person in the employ of the locator assisting by his labor in the performance of such assessment work and knowing the precise situation and boundaries of the mining claim can not make a valid relocation of such mining ground.

Florence-Rae Copper Co. *v.* Kimbel (Washington), 147 Pacific, 881, p. 884, January, 1915.

RELOCATION.

SINKING NEW DISCOVERY SHAFT—APPLICATION OF STATUTE.

That portion of section 7365 of the statute of Washington (Remington and Ballinger Code) that refers to the sinking of a new discovery shaft on a relocated claim does not apply to mining claims west of the summit of the Cascade Mountains.

Florence-Rae Copper Co. *v.* Kimbel (Washington), 147 Pacific, 881, p. 883, January, 1915.

SUFFICIENCY OF NOTICE.

Under the statute of Washington (Remington and Ballinger Code, section 7365) the relocation of a forfeited or abandoned lode claim can only be made by sinking a new discovery shaft and fixing new boundaries, the same as in the original location; or in lieu of the new discovery shaft the relocator may sink the original discovery shaft 10 feet deeper and erect new or renew the old monuments, and the relocation certificate must state whether the whole or any part is located as abandoned property; and under this statute a relocation certificate is insufficient if it fails to state that the new location was located as "abandoned property." The fact that the mining claim had been forfeited does not relieve the relocator from the necessity of stating in his certificate that the ground was located as abandoned property, as the words "abandoned" and "forfeiture" in the statute are used synonymously and interchangeably, and the word "abandoned" in the latter part of the section includes both the terms "forfeiture" and "abandoned" as used in the first part of the section.

Florence-Rae Copper Co. *v.* Kimbel (Washington), 147 Pacific, 881, p. 883, January, 1914.
See Clason *v.* Matko, 223 U. S., 646.

SUFFICIENCY OF POSTED NOTICE.

The provision of section 7365 of the statutes of Washington (Remington and Ballinger Code), to the effect that a location certificate shall contain a statement as to whether the ground is located in whole or in part upon forfeited or abandoned ground, obviously and necessarily refers to the notice to be posted upon the ground, and a posted notice that fails to contain this statement is insufficient.

Florence-Rae Copper Co. *v.* Kimbel (Washington), 147 Pacific, 881, p. 884, January, 1915.

BURDEN OF PROOF—PERFORMANCE OF ASSESSMENT WORK.

Where a valid mining location is made the title thus acquired remains so whether the annual assessment work is performed or not, until forfeited or abandoned; and a party seeking to initiate a claim to the mining premises already legally located must prove that the annual labor thereon has not been performed in order to establish the fact that the ground so located is subject to relocation.

Lancaster *v.* Coale (Colorado Appeals), 150 Pacific, 821, July, 1915.

GROUND LOCATED AS ABANDONED PROPERTY.

While mineral lands revert to the public domain by abandonment or forfeiture of a valid location, yet its character is so changed by the original location so that after such reversion it can not be relocated as other mineral lands of the public domain, but requires a different form of location notice, stating a fact, preserving this peculiar character derived from the prior location, and giving notice to the world that the ground was of that character of the public domain when the subsequent location was commenced.

Copper Queen Consolidated Mining Co. *v.* Stratton (Arizona), 149 Pacific, 389, p. 391, June, 1915.

RELOCATION ON ABANDONED OR FORFEITED GROUND.

The only restraint the statute of Arizona (Civil Code, 1901, paragraph 3241) places upon the relocation of mineral lands after an original location has been made is such as prevents the relocator, by his relocation, from depriving the former locator of any of his rights; but where all the rights of the original locator have been abandoned or forfeited and no claim of such right is asserted by any proper person, and the contesting parties concede that all rights existing by reason of such prior location ceased to exist before the attempted relocation, then the ground was open to relocation in the same manner as if no location had ever been made, and the ground at such time was in character open, unappropriated public mineral land subject to location in the same manner as if it had never been located, and, being of such character, a location made in such manner as an original location is required to be made was effective as such, and its validity can not be questioned upon the same grounds that a location upon ground that had theretofore been the subject of location can be questioned; and a valid location of such ground can be made without the locator stating whether the whole or any part thereof was located as abandoned property, as such locator or relocator was not in fact locating the ground as abandoned property, but was locating it in the same manner as other public mineral ground is located—that is, as an original location; and to complete such an original location the law does not require the locator to state in his location notice whether the whole or any part of the location was located as abandoned property.

Copper Queen Consolidated Mining Co. *v.* Stratton (Arizona), 149 Pacific, 389, p. 392, June, 1915.

POSSESSORY RIGHTS.

ACQUIRED BY VIRTUE OF LOCATION.

The title and rights acquired by virtue of a valid location of a lode mining claim upon the public domain are subject to the paramount title of the United States to the lands upon which such claims may be located, and such right may be forfeited by a failure to perform conditions imposed by the Government for holding property; but this possessory right, with the enjoyment incident thereto, called a mining claim, is, after the acquisition of the title to the land on which is located by a patent from the Government, called a mine.

Earhart *v.* Powers (Arizona), 148 Pacific, 286, p. 287, May, 1915.

CONDITIONS—PERFORMANCE OF ASSESSMENT WORK.

The right of continuous occupation of a mining claim properly located under the statute may be maintained by keeping up the assessment work prescribed by law; and this may be done without incurring the obligation toward the Government of buying and paying for the same; and when a person entitled to the benefit of the statute has made a location in accordance therewith and has gone into possession of the same, he is said to be the owner and in the possession of the mining claim thus located.

Trinity Gold Dredging & Hydraulic Co. *v.* Beaudry, 223 Federal, 739, p. 741, May, 1915.

POSSESSION—PARAMOUNT TITLE.

Congress has by statute provided for the existence of an exclusive right to the possession of a mining claim, while the paramount title to the land remains in the United States, and this right of possession is made separable from the fee and a perfected location has the effect of a grant by the United States of the right of present and exclusive possession.

Trinity Gold Dredging & Hydraulic Co. *v.* Beaudry, 223 Federal, 739, p. 742, May, 1915.
See United States Mining Statutes Annotated, 114.

SALE AND TRANSFER.

MODE OF CONVEYANCE.

The usual mode of conveyance of a mining claim is by deed.

Trinity Gold Dredging & Hydraulic Co. *v.* Beaudry, 223 Federal, 739, p. 741, May, 1915.
See United States Mining Statutes Annotated, 124, 188.

AGREEMENT TO CONVEY—POSSESSORY TITLE.

An agreement by which one party is given the option to purchase certain mines and mining claims designated by name, of which a certain number were described as patented, another certain number as unpatented, and others as having receiver's receipts issued, must be construed as dealing with the possessory title as to all unpatented claims and as binding the obligor to convey his possessory title and right, especially in view of the fact that the purchasing party was to pay all expenses, fees, and costs in connection with contests in the land office and was to perform the annual assessment work and improvements on all unpatented claims; and a stipulation that the final conveyance should be by deed was intended that the conveyance should be of the mining claims as such, and not of the ultimate title to the land known to the parties to rest in the General Government.

Trinity Gold Dredging & Hydraulic Co. *v.* Beaudry, 223 Federal, 739, p. 742, May, 1915.

ADVERSE CLAIMS.

FAILURE TO FILE—NO OBJECTION TO APPLICATION.

The issuance of a patent for a mining claim can not be stayed by reason of some one else claiming a better right to the possession of the claim, unless the person making such claim files the same against the application in the land office; and an action brought in support of such adverse claim must be based on the right asserted in such claim, as it must be conclusively assumed that no adverse claim exists except such as have been filed.

Lancaster *v.* Coale (Colorado Appeals), 150 Pacific, 821, p. 822, July, 1915.

PLACER CLAIMS.

RULE OF APPROXIMATION.

The rule of approximation is now applicable to placer mining locations and entries upon surveyed lands, to be applied on the basis of 10-acre legal subdivisions.

McKitrick Oil Co., In re Land Decisions, August 13, 1915.

LOCATIONS BY INDIVIDUALS FOR CORPORATION.

Where placer mining claims were located by a number of persons with the understanding that each of such locators would have an equal interest in all of the land so located, and where it was the intention and understanding of such locators that a corporation would be organized by them for the purpose of developing the claims and that to such company when organized the claims would be conveyed, the stock of the corporation distributed among such persons according to their respective interests in the land to be conveyed, and where such persons subsequently met, organized a corporation under the laws of the State, and subscribed stock in proportion to the amount and value of the land located by each, such locations are held to be valid, as the locators under such circumstances located the claim solely for their own individual benefit and not as mere agents for the benefit of some other person or of some corporation in which they had no interest; and under their arrangement the corporation to which it was proposed to transfer the claims was to be one in which such locators were to be the sole stockholders and each the owner of the equal undivided part of the stock; and it is not a case of dummy locators lending their names to persons or corporations for the purpose of permitting them to acquire lands.

McKitrick Oil Co., In re Land Decisions, August 13, 1915.

PATENTS.

APPLICATION AND RIGHT OF LOCATOR AS AGAINST THIRD PERSONS.

Where the right to a patent to a mining claim from the United States is the issue and the purpose of the suit, and the evidence is sufficient to satisfy the requirements of the statute and entitle the party, by reason of prior location, to a patent, it would be sufficient to sustain the verdict and judgment, so far as the rights of all the parties are concerned, and the question of title in a third person or a third person's interest can not be raised to defeat the right to a patent from the United States to a mining claim.

Lancaster *v.* Coale (Colorado Appeals), 150 Pacific, 821, p. 822, July, 1915.

DEPARTMENTAL DETERMINATION—NOT SUBJECT TO COLLATERAL ATTACK.

The Land Department of the United States, including in that term the Secretary of the Interior, the Commissioner of the General Land Office, and other subordinate officers, constitutes a special tribunal, vested with judicial power to hear and determine the claims of all parties to the public lands which it is authorized to dispose of and with power to execute its judgments by conveyances to the parties entitled to them; and a patent to land within its jurisdiction, issued by the Land Department, is the judgment of that tribunal and a conveyance of the legal title to the land to the patentee is an execution of the judgment; and when a patent is issued it is, like the judgments of other judicial tribunals, impervious to collateral attack. Accordingly, a decision by the Secretary of the Interior, on appeal from the Land Office, to the effect that a prior placer entry on Government land opened to entry was invalid, as to the extent of any existing conflict with the homestead entry, for the reason that the land was not mineral in character and a cancellation of such entry and the directing the issuance of a patent to the homestead claimant must be regarded by the courts as a judicial determination of an issue within the jurisdiction of such special tribunal, vested with judicial powers to hear and determine matters in controversy, and a patent issued on such determination is not subject to collateral attack in an action brought by the homestead claimant against the placer entryman to recover possession of the disputed ground.

Olson *v.* Kirk (South Dakota), 153 Northwestern, 893, July, 1915.
See King *v.* McAndrews, 111 Federal, 860.
United States Mining Statutes Annotated, 296, 316, 1135.

MINING PARTNERSHIPS.

JOINT OWNERSHIP AND OPERATION.

Under the Civil Code of California (section 2511), a mining partnership exists when two or more persons who own or acquire a mining claim for the purpose of working it and extracting the minerals therefrom actually engage in working the same; but the actual working of the mine by the joint owners is essential to the mining partnership.

Peterson *v.* Beggs (California Appeals), 148 Pacific, 541, p. 542, March, 1915.

JOINT VENTURE AND OPERATION.

Persons who enter into a joint venture for the purchase and operation of mining property and who understand and act upon the understanding that each of the parties shall pay an equal amount of all expenses incident to the venture, likewise understanding that they would share equally in the proceeds of the enterprise, are partners and the arrangement constitutes a partnership.

Galbraith *v.* Develin (Washington), 148 Pacific, 589, p. 591, May, 1915.

AGREEMENT TO ACQUIRE AND DEVELOP MINES.

An agreement among certain persons creating an association of the parties for the purpose of acquiring, developing, and dealing in mines would not create a mining partnership, unless it further provided that when the mining property was acquired and developed it should then be worked on general account.

Peterson *v.* Beggs (California Appeals), 148 Pacific, 541, p. 542, March, 1915.

WHAT CONSTITUTES—COOPERATION IN WORKING MINE ESSENTIAL.

The actual working of a mine by the owners together for their mutual benefit seems to be essential to the existence of the partnership relation, the parties to contribute to the expenses of the work and to share in the profits according to their respective interests, and the partnership arises only when the coowners unite and cooperate in working the mine.

Peterson *v.* Beggs (California Appeals), 148 Pacific, 541, p. 542, March, 1915.

VERBAL AGREEMENT AS TO JOINT VENDOR—PERFORMANCE—STATUTE OF FRAUDS.

An agreement between three persons to procure an option to further develop a mine and to form a corporation and issue stock, to be equally divided among them, while not in writing, amounted to a joint venture or partnership and is not within the statute of frauds; and the procurement of the option, the formation of the corporation, and the issuance of the stock is such performance as to render the statute of frauds inapplicable, even if the original agreement had been void under the statute.

Kent *v.* Costin (Minnesota), 153 Northwestern, 874, p. 875, July, 1915.

CONTRACT INSUFFICIENT TO CREATE.

A contract by which one of the parties holding the legal title to certain mines and mining properties in trust for all was to sell such property or any portion of it and from the purchase price from time to time deduct a sum sufficient to repay him for all sums paid out by him for the benefit of the mining properties and to compensate him for his personal services, the sums remaining to be divided equally among the parties, does not constitute a mining partnership.

Peterson *v.* Beggs (California Appeals), 148 Pacific, 541, p. 542, March, 1915.

MINE WORKED BY ONE COOWNER.

Where one coowner of mining property engages in working it for ore, the remaining owners or cotenants not so engaged do not thus become partners but will be left to their rights and are chargeable according to their duties as cotenants only.

Peterson *v.* Beggs (California Appeals), 148 Pacific, 541, p. 542, March, 1915.

COTENANT SEPARATELY WORKING MINES.

Where a joint owner of mines and mining property who held the title in trust for all the owners under an agreement by which he was to sell the mining property and from the purchase price deduct sums sufficient to pay the expenses incurred, engaged in working the mine and himself compensated the laborers employed, and where in his absence another one of the joint owners took possession and worked the mine, this does not constitute a partnership, and neither of the coowners is liable for espenses incurred by the other.

Peterson *v.* Beggs (California Appeals), 148 Pacific, 541, p. 542, March, 1915.

ONE PARTNER PURCHASING INTEREST OF ANOTHER—FRAUD AND CONCEALMENT—LIABILITY.

Good faith requires that every mining partner in the purchase and disposal of mining property should not make any false representations to his partners as to the sale and purchase of the interests of other partners, and likewise he should abstain from all concealments which may be injurious to the partnership business; and if any such partner is guilty of concealment and derives a profit and benefit therefrom, he may be compelled in equity to account to the other partners; and if in the purchase by one partner of the interests of others and in the purchase misrepresented and concealed the conditions existing at the time and subsequently sold the interest so purchased at a much larger sum, he may in equity be required to account for the excess received by him.

Galbraith *v.* Develin (Washington), 148 Pacific, 589, p. 592, May, 1915.

RIGHTS OF PARTNERS TO BUY AND SELL.

Any one of several partners engaged in the purchase and operation of mining properties has the right to purchase the interest of any of the other partners and acquire the same for himself, and if he subsequently sells the interests so purchased at considerable profit, without any previous arrangement made therefor, his selling partners have no right to complain.

Galbraith *v.* Develin (Washington), 148 Pacific, 589, p. 591, May, 1915.

STATUTES RELATING TO MINING OPERATIONS.

CONSTRUCTION, VALIDITY, AND EFFECT.

PURPOSE AND APPLICATION OF ACT REGULATING COAL MINING.

The manifest purpose of the act of April 18, 1911 (Acts of Alabama, 1911, p. 500), is to reduce to a minimum the dangers incident to the hazardous occupation of mining coal and thus conserve the life and limbs of those engaged in this occupation. Its provisions are not only for the benefit of those occupying the relation of servants or employees but for the protection of all persons engaged in working in mines, whether under contract as an employee or independent contractor.

Stith Coal Co. *v.* Harris (Alabama), 68 Southern, 797, p. 798, May, 1915.

EXAMINATION AND CLASSIFICATION—MANDAMUS TO COMPEL EXAMINING BOARD TO ISSUE CERTIFICATE.

The act of Pennsylvania of June 8, 1901, provides that every applicant for a certificate as mine inspector must have knowledge of the different systems of work in coal mines and must produce satisfactory evidence to the board of examiners of experience in mines where noxious and explosive gases are evolved, and must have had at least five years' practical experience in the anthracite mines of Pennsylvania; but, in addition to this, the board of examiners may exercise discretion in performing their duty, and at least four members of the board must be convinced that an applicant has passed a satisfactory examination before he is entitled to a certificate of qualification; and if the board fails to agree that a particular applicant has passed "a successful examination" and four members are not willing to sign the certificate, the matter there ends, and the applicant can not by mandamus compel the board to issue a certificate, especially where his petition makes no averment as to fraud or misconduct in the refusal to award him a certificate and where he fails to allege that he correctly answered 90 per cent of the questions stated in his written examination.

Reese *v.* Pollard (Pennsylvania), 94 Atlantic, 246, p. 247, March, 1915.

STATUTE REQUIRING OPERATOR TO FURNISH PROPS—REASONABLE COMPLIANCE.

While the statute of Alabama (section 38, act of April 18, 1911) imposing the duty on the mine operator to furnish props to a miner on proper request is mandatory and places an imperative duty upon the owner or operator of a mine and should be construed that the ends for which it was intended may be accomplished, yet it must receive a reasonable construction, and its purpose is to have furnished at a convenient place a sufficient supply of props and other timbers useful for propping in the mine and of suitable lengths and sizes when needed and requested by the miners; but there is nothing in the section indicating that the mine operator is required to furnish props of the exact length demanded by a miner, but the demands of the statute are complied with where the operator furnishes props entirely suitable for the purpose for which they were ordered and which require no unusual effort or inconvenience in setting them, although they may vary an inch from the exact length ordered; and evidence is admissible to show that props an inch or two inches shorter or an inch or two inches longer than the length requested by the miner are suitable in connection with the methods of mining and the particular conditions of the mine where the props are to be used and the circumstances surrounding their use.

Stith Coal Co. *v.* Sanford (Alabama), 68 Southern, 990, p. 992, May, 1915.

SCOPE OF EMPLOYMENT—MINER WITHIN PROTECTION OF STATUTE.

Where all the men employed in a mine, including the boss driver and his assistant, are subordinate to the bank boss and subject to his orders and control; and where in case of a wreck of trip cars in a mine the bank boss ordered all persons within hearing to assist in cleaning up the wreck and getting the cars running, and the boss driver being present thereupon ordered his assistant boss driver to aid in cleaning up the wreck, such assistant boss driver is then within the protection of the statute protecting employees against the negligence of an employer, though the particular service is foreign to his regular work.

Republic Iron & Steel Co. *v.* Quinton (Alabama), 69 Southern, 604, p. 605, July, 1915.

AMENDATORY ACT INVALID—EFFECT.

The act of 1907, chapter 540, attempting to amend chapter 237 of the acts of 1903, of the Tennessee statutes, declaring that the mine foreman should be considered the agent and representative of the owner or operator in the discharge of the statutory duties of such foreman, being invalid by reason of the failure of the legislature to observe the constitutional requirements in the manner of its passage, leaves the provisions of the original act in force, and under such original act a mine owner or operator was not liable to a miner injured by the negligence of such statutory foreman.

Tennesse Coal, Iron & Railroad Co. *v.* Hooper (Tennessee), 175 Southwestern, 1146, May, 1915.

See Roame Iron Co. *v.* Francis, 130 Tennessee, 694; 172 Southwestern, 816.

EMPLOYERS' LIABILITY ACT—APPLICATION AND LIABILITY.

In an action for damages for injuries by a miner under the employers' liability act of Alabama (code section 3910), the injured miner can not recover without showing the relation of employee to the defendant operator, as this section imposes no duty upon a mine operator to any but his employees.

Stith Coal Co. *v.* Harris (Alabama), 68 Southern, 797, p. 798, May, 1915.

EXERCISE OF CARE

The statute of Missouri (Revised Statutes of 1909, section 7828), requiring certain machinery dangerous to employees when engaged in their ordinary duties to be guarded, does not exempt the employees from the duty to exercise reasonable care to avoid injury from such machinery. Nor does it deprive the employer of the defense of contributory negligence to actions for injuries caused thereby.

Northern Central Coal Co. *v.* Hughes, 224 Federal, 57, p. 59, May, 1915.

DUTIES IMPOSED ON OPERATOR.

DUTY TO PROVIDE SAFE APPLIANCES.

Under the statute of West Virginia it is the nonassignable duty of the owner or operator of a coal mine, and not of his foreman, to maintain the motor tracks, motor, and other appliances with which the miners are required to work in a reasonably safe condition, and for any failure in this respect resulting in an injury to a miner, such owner or operator is liable for actionable negligence.

Gray *v.* Pocahontas Consolidated Collieries Co. (West Virginia), 85 Southeastern, 551, p. 552, May, 1915.

DUTY TO FURNISH PROPS—COMPLIANCE AS TO LENGTH OF PROPS.

An action by a miner injured by a fall of rock from the roof of a mine in his working place, based on the alleged violation by the mine operator of the statute of Alabama (section 38, act of April 18, 1911), imposing upon the operator the duty of furnishing needed props or timbers when properly requested by the miner, can not be maintained, and a recovery can not be had where it appears from the evidence that the vein of coal in the room where the rock fell was 3 feet and 5 inches high, and where on demand the operator furnished the miner with props 3 feet and 4 inches, 3 feet and 6 inches, and 3 feet and 8 and 10 inches, and where the evidence shows that a prop 3 feet and 4 inches and props 3 feet and 6 inches are suitable and proper to be used in propping the roof of a mine where the vein of coal is 3 feet and 5 inches high, though the operator did not in fact furnish props cut 3 feet and 5 inches; and on the trial of the case it was proper for the operator to show that for coal veins of the alleged thickness a prop 3 feet and 4 inches was suitable because such props were ordinarily used with a cap board, an instrument in general use in mines, and about 4 or 5 inches wide, about 12 inches long, and about 1 inch thick, and that if a prop is a little short of the required length, the use of such cap board in connection with the props is a better method of propping the roof of a mine; and it was also proper for the operator to show that a prop 3 feet and 6 inches was suitable and was used in well regulated mines for propping purposes in veins of the alleged thickness, and that such props were set, under such circumstances, by digging an inch or more into the floor of the mine, and that the furnishing of such props was a compliance with the imposed statutory duty.

Stith Coal Co. *v.* Sanford (Alabama), 68 Southern, 990, p. 991, May, 1915.

APPLIANCES—ABSENCE OF PROOF OF NOTICE OF DEFECTS.

A coal-mine operator is not liable under the Alabama employers' liability act (section 3910, subdivision 1, Code of 1907), to an employee employed as a coupler on a motor car used for hauling trips of cars out of the mine where, at the time of the injury complained of, the plaintiff was riding on a motor car, engaged in the line of his employment in coupling the power car to a trip of coal cars to be hauled out of the mine, and where the averments of negligence relied upon are to the effect that the bumpers on the tram car that caught the plaintiff's hand and inflicted the injury complained of were "broken off," and where such fact is averred as constituting the defect in the ways, works, machinery, or plant, of the defendant that was the proximate cause of the injury, and had not been discovered or remedied owing to the negligence of the defendant, and where it appears from the evidence that the car had had bumpers and where there was nothing to show for what length of time immediately before the accident the bumpers had been broken off, as the bumpers must have been broken off the car a sufficient length of time to charge the mine operator with negligence in failing to discover the defect and remedy it, in the absence of actual knowledge of the defect.

Sloss-Sheffield Steel & Iron Co. *v.* Westbrook (Alabama), 69 Southern, 311, June, 1915.

VIOLATION OF STATUTORY DUTIES—LIABILITY.

PLEADING NEGLIGENCE—SUFFICIENCY OF COMPLAINT FOR INJURY.

In an action by a miner for damages for injuries he must, under the Pennsylvania act of May 25, 1887, aver facts sufficient to show that a duty required by law has been breached or neglected by the mine operator, and the averments must be sufficient to indicate the causal connection between such breach or neglect and the injury complained of and must be sufficient to give the defendant clear and exact information of the charges against him; and while he need not anticipate defenses or aver mere underlying evidentiary facts, yet the elementary facts relied upon to show negligence must appear in unequivocal language and these facts must be such as, standing alone, if not controverted, would entitle the plaintiff to a verdict in his favor; and if many separate grounds of complaint are laid in a declaration each must be examined with like care, and a multitude of charges, none of which aver facts sufficient to make a case against the defendant, are no stronger than one insufficient averment would be.

Charnogursky *v.* Price-Pancoast Coal Co. (Pennsylvania), 94 Atlantic, 451, p. 452, March, 1915.

A complaint in an action by a miner for personal injuries is sufficient where it avers that the injuries complained of were received by reason and as a proximate consequence of the negligence of an employee of the defendant operator, and intrusted by the defendant with superintendence while in the exercise of such superintendence, in this, that such person negligently permitted the top or roof of the mine where plaintiff was so engaged in the discharge of his duties to be defective or to be improperly supported where plaintiff was at work; or negligently permitted plaintiff to work in a dangerous part of such mine, where rock was liable to fall on plaintiff, without warning or notifying him of such danger, as it sufficiently shows a relation between the parties out of which arises a duty owing from the operator to the plaintiff, and it sufficiently avers that the operator negligently failed to do and perform the act or acts imposed by that duty; and it is not necessary and the plaintiff is not required to define the quo modo or to specify the particular acts of diligence which should have been performed in the discharge of the duty, and what the defendant did and how he did it and what he failed and omitted to do are generally better known to him than to the plaintiff; neither was it necessary to aver that the defendant knew of the alleged defective condition of the top or roof of the mine.

Tennessee Coal, Iron & Railroad Co. *v.* Moore (Alabama), 69 Southern, 540, June, 1915.

The duty of the operator to warn and the knowledge of the operator, under the superintendent subdivision of the Alabama statute, is embraced in the words "negligently caused," "negligently allowed," "negligently permitted," and "negligently failed to warn"; and where a complaint by a miner for injuries caused by the fall of a roof charges the operator with negligent failure to properly support the roof, or that the operator negligently permitted it to be and remain defective, or that the operator negligently permitted plaintiff to work in the dangerous place without warning him of the dangers, are sufficient allegations of the negligent failure of the operator, through its superintendent, to discharge such duty to the plaintiff.

Tennessee Coal, Iron & Railroad Co. *v.* Moore (Alabama), 69 Southern, 540, p. 541; June, 1915.

EMPLOYERS' LIABILITY ACT—NEGLIGENT ORDER GIVEN BY OPERATOR—BURDEN OF PROOF.

In an action under the employers' liability act of Alabama (Code of 1907, section 3910, subdivision 3), by an experienced miner for injuries received while working in defendant's mine by a rock falling from the roof, due to the alleged negligence of the superintendent of the defendant operator in ordering the complainant to put an entry in which he was working on center, the burden of proof is on the injured miner to show that the order, in the execution of which he was injured, was a negligent order under the circumstances and that it must have been reasonably apparent to the superintendent, under the condition as he knew or ought to have known it, that the miner's execution of his command would expose the miner to some peril beyond the ordinary risks of his service, and against which ordinary and reasonable care on his part would probably not suffice to protect him.

Woodward Iron Co. *v.* Wade (Alabama), 68 Southern, 1008, p. 1010, June, 1915.

FAILURE TO FURNISH PROPS—LIABILITY.

A complaint in an action by an injured miner against a mine operator for damages for injuries received while mining coal in a room in the defendant's mine, caused by a rock falling upon the miner from the roof, is sufficient where it further avers that the miner demanded and requested of the person whose duty it was to deliver or have delivered certain props or timbers needed in the mine, and that it was the duty of the defendant operator to promptly deliver the props as requested and demanded, and that as the proximate consequence of such failure the miner was injured.

Stith Coal Co. *v.* Sanford (Alabama), 68 Southern, 990, May, 1915.

ORDERING MINER INTO DANGEROUS PLACE—FAILURE TO WARN.

Where a miner is directed by the mine operator to conduct mining operations in a place in the mine that is dangerous, and where the danger or peril is not obvious, but is inherent in the conditions necessarily surrounding the miner while executing the order of the operator, and where the conditions are such that the operator could and should have known of the danger, and of which, if not remedied, the miner could expect the operator to seasonably inform him, then in such case the operator's order without such warning is negligent and the miner may recover for an injury proximately resulting therefrom under the employers' liability act of Alabama.

Woodward Iron Co. *v.* Wade (Alabama), 68 Southern, 1008, p. 1010, June, 1915.

FAILURE TO INSPECT MINE.

Under section 5006, General Statutes of Kansas, 1909, it is the duty of mine operators whose mines generate fire damp to keep them free from standing gas, and to examine every working place carefully each morning with a safety lamp by an examiner or fire boss before miners are permitted to enter their working places; and a failure to make such inspection of a mine shown to have generated fire damp or gas will render the operator liable to a miner injured thereby.

Hartman *v.* Dickinson (Kansas), 148 Pacific, 743, May, 1915.

EMPLOYMENT OF MINOR.

In an action in trespass to recover damages for physical injuries to a boy employed in a coal operator's coal breaker where the petition averred that the injuries complained of resulted from the defendant's violation of duties and regulations prescribed by the laws of the Commonwealth for the safety of laborers and employees, and showing by averment the boy's age and that he was injured while in the service of the defendant company, is sufficient to show a liability and to sustain a recovery grounded on the employment of a minor under the age of 16 in violation of the act of May 1, 1909, though the better practice would have been to aver the specific violation of the act.

Krutlies *v.* Bulls Head Coal Co. (Pennsylvania), 94 Atlantic, 459, April, 1915.

UNLAWFUL EMPLOYMENT OF MINOR—EVIDENCE OF NEGLIGENCE—STATUTORY DEFENSE—PLEADING.

When the employment of a minor is shown to be illegal, because forbidden by a statute, this in itself is sufficient evidence of the defendant's negligence, and where the injury complained of occurred in the course of the plaintiff's service under such unlawful employment, that is enough to show causal connection, and the law will refer the injury to the original wrong as its proximate cause. To employ a minor under the age of 16 in a coal breaker is made unlawful by the statute of Pennsylvania, unless the employer procures and keeps on file the certificate required by the act, and when an action for damages is brought grounded on such employment, a procurement of the certificate and a copy of the same on file as required by the act is a matter of justification or defense to be shown by the employer, and as a lack of compliance with the terms of the statute in this respect on the part of the defendant need not be shown by the injured minor, it is therefore not required to be averred in his statement of claim.

Krutlies *v*. Bulls Head Coal Co. (Pennsylvania), 94 Atlantic, 459, p. 460, April, 1915.

EMPLOYMENT OF BOY UNDER 16—FALSE REPRESENTATION AS TO AGE—LIABILITY.

A mine operator employing a boy under 16 years of age to work in its coal breakers and who fails to procure the certificate and keep the same on file as required by the act of May 1, 1909, is liable in damages for an injury sustained, though the boy falsely represented his age at the time of the employment, as a false statement by the boy in regard to his age will in no sense bar a recovery for subsequent injuries.

Krutlies *v*. Bulls Head Coal Co. (Pennsylvania) 94 Atlantic. 459, p. 461, April, 1915.

EMPLOYMENT OF MINOR—COMPLIANCE WITH STATUTE—PROCURING AND FILING CERTIFICATE.

Under the Pennsylvania statute of May 1, 1909 (P. L. 375), a coal operator who employs in his coal breakers a minor under 16 years of age does so at his own risk, so far as the question of age may enter into a subsequent liability, unless he shows compliance with the act and has obtained and kept on file a certificate as required by the act.

Krutlies *v*. Bulls Head Coal Co. (Pennsylvania). 94 Atlantic, 459, p. 460, April, 1915.

EMPLOYMENT OF YOUTHFUL MINER NOT NEGLIGENCE.

Under the statute of West Virginia it is not negligence per se for a mine operator to employ a boy over 14 years of age in a coal mine, but, on the contrary, the statute permits such employment.

Gray *v*. Pocahontas Consolidated Collieries Co. (West Virginia). 85 Southeastern, 551, p. 552, May, 1915.

LIABILITY FOR INJURIES TO EMPLOYEE OF INDEPENDENT CONTRACTOR.

The act of Alabama regulating the mining of coal (act of 1911, 500, p. 514) is for the benefit of miners, whether an employee of the mine operator or of an independent contractor, and if, as a proximate consequence of a breach of duty imposed by the statute on a mine operator a miner is injured, he is entitled to recover; and it is immaterial in this case whether he was an employee of the owner and operator, or an independent contractor, or an employee of an independent contractor, and any averments in his complaint as to his relation to the mine owner and operator will not require strict proof.

Stitch Coal Co. *v.* Harris (Alabama), 68 Southern, 797, p. 798, May, 1915.

DUTIES IMPOSED ON MINER.

FAILURE TO SELECT AND MARK PROPS—PROOF OF CUSTOM.

Section 2739b of the statutes of Kentucky makes it the duty of a mine owner to furnish props and caps to the miner only after the miner has selected and marked the same, and the statutory duty thus imposed on the miner can not be abrogated by a custom of merely requesting props and caps.

Peerless Coal Co. *v.* Taylor (Kentucky), 176 Southwestern, 1002, p. 1003, June, 1915.

See Palmer *v.* Empire Coal & Coke Co. (Kentucky), 162 Ky. 130, 172 Southwestern, 97.

SELECTION OF PROPS—FAILURE OF OPERATOR TO PROVIDE PROPS.

Under section 2739b of the statutes of Kentucky, making it the duty of a mine owner to furnish props and caps only after the miner has selected and marked the same, if the mine owner fails to supply props at a place in the mine, or within a reasonable distance therefrom, so that they can be conveniently reached by the miner, then the miner is not required to select and mark the props, but a mere request on his part will be sufficient to impose on the owner the duty of furnishing props, and the failure of the mine owner or operator to so furnish the props may make him liable for an injury to the miner resulting proximately from such failure.

Peerless Coal Co. *v.* Copenhaver (Kentucky), 176 Southwestern, 1002, p. 1003, June, 1915.

DUTY OF MINER TO AVOID PERILS—ASSUMPTION OF COMPETENCY BY OPERATOR.

Where an experienced miner is directed to conduct mining operations at a particular and perilous place in the mine, and where the peril is obvious to the miner and might readily be avoided by him by fully discharging his duty in conformity with the order given, the operator has the right to assume that the miner will both observe the peril and avoid it, and under such circumstances it can not be said that the order was negligently given within the employers' liability act of Alabama.

Woodward Iron Co. *v.* Wade (Alabama), 68 Southern, 1008, p. 1010, June, 1915.

NEGLIGENCE OF MINE SUPERINTENDENT, FOREMAN, OR BOSS.

LIABILITY WHERE MINE OPERATIONS ARE PLACED IN THE HANDS OF STATUTORY FOREMAN.

Under a statute requiring a mine operator to employ a statutory mine foreman and depriving the owner or operator of any control of such foreman as to the internal operations of the mine, the relation of master and servant does not exist, and it would be unreasonable and against conscience to hold the owner or operator responsible for the consequences of an act of such foreman, the doing of which had been, by the express provision of the statute, taken out of his hands and placed beyond his control.

Tennessee Coal, Iron & Railroad Co. *v.* Hooper (Tennessee), 175 Southwestern, 1146, May, 1915.

NO LIABILITY IN ABSENCE OF RELATION OF MASTER AND SERVANT.

A mine foreman under the Tennessee statute (Acts of 1903, ch. 237) is charged with the duty of keeping watch over entries and traveling ways so that as the miners advance their excavations all dangerous coal, slate, or rock overhead is taken down or secured against falling, and the statute prescribing the qualifications of mine foremen compels the operator to employ them and expressly provides that such foremen shall not be subject to the control of the operator or owner in the discharge of their statutory duties and the performance of these duties is secured by the imposition of a fine and imprisonment upon any foreman neglecting them; and by reason of this statutory provision the relation of master and servant does not exist between the foreman and the mine owner or operator, and the owner or operator is not liable to an injured miner by reason of the negligence of such a statutory foreman.

Tennessee Coal, Iron & Railroad Co. *v.* Hooper (Tennessee), 175 Southwestern, 1146, May, 1915.

REQUIRING EXPLOSIVES TO BE KEPT IN DAMP PLACE.

A fire boss has no right under the Pennsylvania statute to say that a miner's box containing explosives should remain in a particular place where it was damp, and where all that he did say was that the place was all right and the box good enough, and the miner could not under such direction leave his box where it was exposed to dampness, relying upon the judgment of the fire boss, and, even if the fire boss had the power and was negligent, the mine owner under the Pennsylvania statute would not be liable for an injury to the miner resulting from the negligence of the statutory fire boss.

Lehigh Valley Coal Co. *v.* Calausky, 222 Federal, 664, p. 666, April, 1915.

NEGLIGENCE OF MINE BOSS—SELECTING DANGEROUS METHODS.

The statute of Indiana, section 8580, Burns's, 1914, makes it the duty of a mine boss to visit and examine every working place in a mine at least every alternate day while the miners are at work and to examine and see that each working place is properly secured by tamping, and shall see that a sufficient supply of timbers are on hand, and shall also see that all loose coal, slate, and rock overhead wherein miners have to travel to and from their work are taken down or carefully secured; and a complaint in an action for damages for the death of a miner which avers that the deceased miner was ordered by the mine boss and his assistant to repair the timber and entry in a particular manner, and that the way selected was dangerous and known to them to be so, but nevertheless they negligently selected the unsafe way of propping the roof when they could have selected the safe way of taking down the loose slate and rock, and that the deceased miner, while obeying the order and directions of the mine boss and his assistant and while attempting to perform the work in obedience to such order, was killed by a fall of the loose slate, is sufficient, for the reason that the statute recognizes two ways of making such places safe; and if the mine boss and his assistant negligently selected the unsafe way, as alleged, and ordered the deceased miner to work in that particular manner, and he lost his life while obeying such order, the mine operator is liable.

Vandalia Coal Co. *v.* Alsopp (Indiana Appellate), 109 Northeastern, 421, p. 422, June, 1915.

EVIDENCE—ADMISSIBILITY OF DECLARATIONS.

Under the Pennsylvania law a fire boss is not a fellow servant of the mine owner's employees, and the employers' liability act does not apply to such a foreman; and where it was not made to appear that a fire boss represented the mine owner in any respect or performed any duties except those imposed upon him by statute, a conversation between him and the injured miner did not affect the mine owner, and is not admissible in evidence in an action by an injured miner.

Lehigh Valley Coal Co. *v.* Calausky, 222 Federal, 664, p. 666, April, 1915.

EFFECT ON CONTRIBUTORY NEGLIGENCE.

DEFENSE OF CONTRIBUTORY NEGLIGENCE NOT ABROGATED.

The statute of Missouri (Revised Statutes of 1909, sec. 7828), requiring certain dangerous machinery used by employees to be guarded, does not deprive the employer of the defense of contributory negligence to actions for injuries caused by a failure to guard dangerous machinery.

Northern Central Coal Co. *v.* Hughes, 224 Federal, 57, p. 59, May, 1915.

CONTRIBUTORY NEGLIGENCE AND ASSUMPTION OF RISK NOT DEFENSES—EXCEPTIONS.

In an action for damages by an injured miner negligence or assumption of risk can not be interposed as a matter of defense under section 3910 of the Code of Alabama, unless the miner is the one whose duty it is to remedy the defect, or who committed the negligent act causing the injury complained of. The statute does not prohibit the defense of contributory negligence or assumption of risk in three distinct classes of cases: (1) Where the miner knows of the defect or negligence and fails to give information thereof within a reasonable time to the operator or to a superior in the service, unless the operator or such superior knows of the defect or negligence; (2) where the injured miner, whose duty it is to remedy the defect alleged to have caused the injury, knew of the existence of the defect or the negligence and thereafter remained or continued in the service and was injured; and (3) where the injured miner himself committed the negligent act causing the injury complained of, and by this meaning a breach of some duty by the miner whereby the defect, or negligence predicable of it, was caused by the act or omission of the servant suffering the injury, thus negativing in that respect the existence of a defense based on assumption of risk or contributory negligence other than may arise from the breach of duty by the miner.

Clinton Mining Co. *v.* Bradford (Alabama), 69 Southern, 4, p. 7, May, 1915.
See Warrior River Coal Co. *v.* Thompson (Alabama), 69 Southern, 76, p. 79, June, 1915.

MINER REMAINING IN EMPLOYMENT—MEANING.

The expression "to remain in the employment" as provided in section 3910, Code of Alabama, with reference to the defenses of contributory negligence and assumption of risk, signifies continuing in the service or work of the mine operator in the zone of possible danger of injury to the miner, who then knows of the existence of the defect or negligence to which under other sections of the act his injury is attributed in his pleading.

Clinton Min. Co. *v.* Bradford (Alabama), 69 Southern, 4, p. 7, May, 1915.

DAMAGES DIMINISHED BY CONTRIBUTORY NEGLIGENCE.

An action by a miner instituted in the State courts of Nevada, or in a Federal court in Nevada, for injuries received by him in the State of California, is governed by the laws of the latter State, and under the statute of California contributory negligence will not bar a recovery where the miner's contributory negligence was slight and that of the mine operator gross in comparison, and in such case the damages may be diminished by the jury in proportion to the amount of negligence attributable to the injured miner.

Keane Wonder Mining Co. *v.* Cunningham, 222 Federal, 821, p. 824, May, 1915.

FAILURE TO TAKE PRECAUTION FOR PROTECTION OF LIFE NOT A DEFENSE—DIMINISHING DAMAGES—INSTRUCTIONS.

A complaint in an action by an injured miner against a coal-mining company averred that the company was engaged in mining coal at such a depth as to render the work inherently dangerous on account of the liability of noxious and combustible gases to accumulate, which could have been guarded against by the exercise of reasonable care and diligence, and that the company failed to install in its mine sufficient ventilating fans and air shafts to dissipate such gases, by reason of which the complainant, employed in the mine, was, on entering a room of the mine as directed by the company, severely burned and injured as the result of a violent explosion occurring at that time, brings the cause of action within the provisions of the statute of Oregon (sec. 6 of the employers' liability act), to the effect that the contributory negligence of the person injured shall not be a defense, but may be taken into account by the jury in fixing the amount of damages, and shows that the mine operator did not comply with section 1 of the statute by using every device, care, and precaution practical to use for the protection and safety of life and limb; and an instruction to the effect that, notwithstanding the negligence or carelessness of the mine operator, if the plaintiff himself has been guilty of contributory negligence, such negligence as proximately contributes to the injury, then the plaintiff can not recover, notwithstanding the negligence of the defendant, is erroneous as not stating the correct rule of law under the statute.

Raiha *v.* Coos Bay Coal & Fuel Co. (Oregon), 149 Pacific, 940, June, 1915.

APPORTIONMENT OF DAMAGES ACCORDING TO WANT OF CARE.

Under the employers' liability act of Oregon (General Laws of Oregon, 1911, chapter 3, section 6) contributory negligence of an injured miner is not a bar to a recovery of damages by a miner for an injury resulting from the alleged negligence of the mine operator, but the measure of the loss occasioned by the injury is apportioned ratably between the parties according to their respective want of ordinary care.

Raiha *v.* Coos Bay Coal & Fuel Co. (Oregon), 151 Pacific, 471, p. 472, September, 1915.

PRIMARY AND CONTRIBUTORY NEGLIGENCE—DISTINCTION A QUESTION OF FACT.

Under a constitutional provision requiring it, contributory negligence is a question which the court must never decide, but it must be submitted to a jury for their consideration; but contributory negligence on the part of a plaintiff presupposes negligence on the part of a defendant, and before the question of contributory negligence on the part of the plaintiff can arise negligence of the defendant must first be shown; and if there is no negligence upon the part of the defendant shown, and the negligence of the plaintiff only, or of his fellow servant, caused the injury, then such negligence is primary and not contributory negligence on the part of the employee, and there is no case to come to the jury where the primary negligence was caused by the plaintiff or by his fellow servant.

Barnsdall Oil Co. *v.* Ohler (Oklahoma), 150 Pacific, 98, p. 103, June, 1915.

ACTION FOR INJURY TO MINER—SUFFICIENCY OF ANSWER PLEADING CONTRIBUTORY NEGLIGENCE.

In an action by a miner for personal injuries occasioned by the alleged negligence of the mine operator under section 3910 of the Alabama Code of 1907, making a mine operator liable for injuries to a miner caused by any defect in the condition of the ways, works, machinery, or plant of the operator, and providing also that it shall not be contributory negligence or assumption of risk on the part of the miner to remain in the employment of the operator after knowledge of the defect or negligence causing the injury, unless it is his duty to remedy the defect or unless he committed the negligent act causing the injury complained of, an answer or a pleading by the operator to the action is insufficient where it avers that the miner was himself guilty of negligence which proximately contributed to the injury and that at the time he sustained the injuries complained of he was engaged in mining in a certain heading in the defendant's mine, in the roof of which near the place at which he was at work there was loose rock or slate likely to fall, which fact and the danger to plaintiff arising therefrom was known, or should have been known to him in the exercise of due care, notwithstanding which plaintiff went under such rock or loose slate and the same fell, striking him and causing the injury complained of, is insufficient for failing to aver that the plaintiff was under the duty to remedy the defect described, or for failing to aver that he himself committed the alleged negligent act causing the injury complained of, as the question of contributory negligence under such circumstances is a defense and must be specially pleaded, under the rule that where there is an exception in a subsequent clause or a subsequent statute it is a matter of defense, to be pleaded and proved by the defendant.

Clinton Mining Co. *v.* Bradford (Alabama), 69 Southern, 4, p. 7, May, 1915.
See Warrior River Coal Co. *v.* Thompson (Alabama), 69 Southern, 76, p. 79, June, 1915.

INJURY FROM FALLING ROCK—ASSURANCES OF SAFETY BY MINE BOSS.

A miner injured by a fall of rock from the roof of an entry at a roadway in a mine is entitled to recover under the Alabama statute (section 3910, subdivision 1, code of 1907) where it appears from the evidence that the miner called the attention of the mine boss to the dangerous condition of the roof under the roadway and the mine boss, after an examination of the roof at the particular place, assured the miner that the place was safe and that supports by means of collars were not required, and where on such assurance the miner continued work and was injured by the fall of rock, and in such case the defense of contributory negligence is not available to the operator.

Warrior River Coal Co. *v.* Thompson (Alabama), 69 Southern, 76, page 80, June, 1915.

EFFECT ON ASSUMPTION OF RISK.

MINER CAN NOT WAIVE VIOLATION OF STATUTE.

It may well be doubted whether a miner in a coal mine can assume the risk incident to a noncompliance with the act of April 18, 1911, of Alabama (Acts of 1911, p. 500), even by a special contract supported by sufficient consideration, and whether such a contract would not be void as contravening the public policy of the State; but it is clear that in the absence of such contract a miner does not assume the risk by continuing at his work, even in violation of rules promulgated by the mine owner for the purpose of evading the statute.

Stith Coal Co. *v.* Harris (Alabama), 68 Southern, 797, p. 798, May, 1915.

ACTION TRANSITORY—STATUTE GOVERNING AS TO ASSUMPTION OF RISK.

An action by a miner for personal injuries is transitory and maintainable wherever a court may be found that has jurisdiction of the parties and the subject matter; and such an action brought in a State court of Nevada upon a cause of action that arose in California, subsequently removed to the United States district court of Nevada, is to be governed by the statute of California, which provides that assumption of risk is no defense to an action of such nature.

Keane Wonder Mining Co. *v.* Cunningham, 222 Federal, 821, p. 824, May, 1915.

MINER'S DUTY TO PROP ROOF—ANSWER OF ASSUMPTION OF RISK.

In an action by a miner for injuries caused by a fall of rock from the roof of an entry or room where he was working, based on the statute of Alabama (section 3910, subdivision 1, Code of 1907), an answer or pleading by the defendant operator is sufficient where it directly avers that the plaintiff himself, on the occasion of which he complained, was the particular miner or servant to whom was entrusted the duty of seeing that the roof of the entry or room at the particular place was in proper condition and that the plaintiff undertook to perform the duty so entrusted to him.

Warrior River Coal Co. *v.* Thompson (Alabama), 69 Southern, 76, p. 79, June, 1915.

LIABILITY OF OPERATOR FOR INJURY TO MINER—ASSURANCES OF SAFETY.

In an action by a miner for injuries caused by a fall of rock from the roof, on the ground of the alleged negligence of the superintendent in negligently causing or permitting the roof to fall upon and injure the miner, the plaintiff is entitled to recover under the employers' liability act of Alabama (section 3910, Code of 1907), abrogating the defenses of assumption of risk and contributary negligence, and especially where there was evidence to show that the rock fell from above a roadway and which roadway ought, for safety, to have been supported by a cross collar, and where it appeared that the injured miner and other miners were prevented from putting in such collars without the order or approval of the mine boss, and where the injured miner had reported to such boss the absence and need of collars at the particularly roadway, and where after the mine boss examined and inspected the place informed the miner that it was a good prop and that collars were not needed.

Warrior River Coal Co. *v.* Thompson (Alabama), 69 Southern, 76 p. 79, June, 1915.

See Clinton Coal Co. *v.* Bradford (Alabama), 69 Southern, 4, May, 1915.

GRATUITOUS SERVICE—SCOPE OF EMPLOYMENT.

There can be no recovery in an action for the death of a person or employee in a mine employed in the capacity of assistant boss driver, who gratuitously, or without proper authorization, undertook to aid in the work of repairing a wreck in the mine, and in so doing handled an electric wire which shocked and killed him.

Republic Iron & Steel Co. *v.* Quinton (Alabama), 69 Southern, 604, p. 605, July, 1915.

STATUTORY ACTION FOR WRONGFUL DEATH.

ELECTION TO ACCEPT UNDER COMPENSATION ACT—DEFENSE.

In an action for the wrongful death of a miner, under the provisions of the statute of Arizona giving a right of action for injuries resulting in death, where there is no averment in the complaint as to whether or not the deceased miner had elected to accept compensation under the provisions of the workmen's compulsory compensation act of Arizona, the fact of such election is a defensive matter; and if not raised by a plea or answer, it is waived by the defendent, the mine operator.

Behringer *v.* Inspiration Consolidated Copper Co. (Arizona), 149 Pacific, 1065, July, 1915.

ACTION UNDER ARIZONA WORKMEN'S COMPULSORY COMPENSATION LAW—EMPLOYERS' LIABILITY ACT—ELECTION OF REMEDIES.

Under the Constitution of Arizona, commanding the legislature to pass a law by which compulsory compensation shall be required to be paid to certain workmen for personal injuries, but leaving it optional with the workmen to settle for such compensation or retain the right to sue as otherwise provided, the legislature does not possess power to enlarge the mandate of the constitution so as to authorize the personal representative of a deceased miner to impose on his heirs and dependents a remedy made by the constitution open to the workman only; but if the workman, prior to his death, should elect to settle for compensation and thereafter die pending the settlement with the employer, his personal representative could enforce the contract; but unless there was an election of this remedy in the lifetime of the deceased miner by him, the personal representative can not maintain an action for compensation, as the employee himself must elect that remedy, and in such case the personal representative is relegated to an action for damages under the statute of Arizona giving a right of action for injuries resulting in death, or an action under the employers' liability act, according as the facts fall within the one or the other of these acts.

Behringer *v.* Inspiration Consolidated Copper Co. (Arizona), 149 Pacific, 1065, p. 1066, July, 1915.

SETTLEMENT PROCURED BY FALSE REPRESENTATIONS.

Where a miner was killed through the negligence of a mine boss in selecting the more dangerous of two ways in attempting to make safe the roof of an entry, a settlement and release of the claim against the mine operator by the widow of the deceased miner is invalid and fraudulent as against an action for damages where the agent and representative of the mine operator falsely and fraudulently represented to such widow that the mine operator had evidence to show that the deceased miner knew the dangers of the roof and went to work under it; that the attorneys representing the widow in an action for damages wholly failed in other similar cases within the knowledge of such agent and that the services of such attorneys were of practically no value, where the agent and representative of the operator knew that at that time she was in great distress on account of the death of her husband and her destitute condition with six small children to support, and further represented that if she went to law and recovered a judgment it would be six or seven years before she would receive anything, and that her lawyers would take most of it if she recovered a judgment, and where before the bringing of the action she repudiated and rescinded the settlement and tendered back the uncashed check.

Vandalia Coal Co. *v.* Alsopp (Indiana Appellate), 109 Northeastern, 421, p. 422, June, 1915.

FAILURE TO USE APPLIANCES TO PREVENT THE ACCUMULATION OF GASES.

It is the duty of a mine operator to use all appliances readily obtainable for the prevention of accidents arising from the accumulation of gas or other explosives, and a failure on the part of the operator to observe that duty constitutes negligence on his part for which he is liable for an injury resulting proximately from such failure.

Raiha *v.* Coos Bay Coal & Fuel Co. (Oregon), 151 Pacific, 471, September, 1915.

MINES AND MINING OPERATIONS.

NEGLIGENCE OR OPERATOR.

NEGLIGENCE DEFINED.

Negligence is the failure to do what a reasonably prudent person would ordinarily have done under the same or similar circumstances, or in doing what a reasonably prudent person under the same or similar circumstances would not have done.

Consolidated Kansas City Smelting & Refining Co. *v.* Schulte (Texas Civil Appeals), 176 Southwestern, 94, p. 95, May, 1915.

FAILURE TO FURNISH PROPS—QUESTION OF FACT.

Where it appeared that a miner marked timbers necessary for his use on the morning of the day before he was injured, and that if the timbers had been promptly delivered the roof from which the rock fell and injured the miner would have been made safe, and that the timbers were not delivered until the afternoon of the next day after they were ordered, and the miner, on starting to move the timbers in place for setting them, was injured by the fall of the roof, it becomes a question of fact for the jury as to whether the operator was guilty of negligence with reference to delivering the timbers and as to whether such negligence was the proximate cause of the injury.

Stith Coal Co. *v.* Harris (Alabama), 68 Southern, 797, p. 799, May, 1915.

In an action by a miner for injuries caused by slate falling from the roof of the room of an entry where he was mining coal, and where there is conflict in the evidence as to whether it was the duty of the miner to prop the roof at the place of injury or to sound the roof and take down the slate if he found it loose, and if the conditions were such that it was his duty to prop the roof, and he would have propped it if props had been furnished, and the mine operator, after the miner had taken proper steps to obtain the props, failed to furnish them, and by reason of such failure the miner was injured, then he would be entitled to recover, unless the danger from lack of props was so obvious and imminent that an ordinarily prudent person would have worked under the circumstances.

Peerless Coal Co. *v.* Copenhaver (Kentucky), 176 Southwestern, 1002, p. 1003, June, 1915.

DUTY TO KEEP ROOF SAFE CAN NOT BE DELEGATED.

A mine owner and operator can not relieve himself of liability by committing to an independent contractor the performance of a specific duty it owes a miner employed by such independent contractor to maintain the roof of an entry in a reasonably safe condition; and whether an alleged independent contractor was such or whether he was an employee of the owner and operator, the owner and operator would be liable for an injury to an employee if, as a proximate consequence of his negligence in leaving the roof in a dangerous condition, it resulted in the injury to the miner, as the person upon whom a positive duty is imposed by law can not delegate in any manner the performance of that duty so as to relieve himself from responsibility for the condition thus created, and the duty and the responsibility rest where the duty is imposed, and a principal is liable for any injury that arises from the nonperformance of the duty or in consequence of it having been negligently performed either by himself or by an independent contractor.

Sloss-Sheffield Steel & Iron Co. *v.* Hubbard (Alabama), 68 Southern, 571, p. 572, May, 1915.
Stith Coal Co. *v.* Harris (Alabama), 68 Southern, 797, p. 798, May, 1915.

FAILURE TO INSTRUCT—MASTER'S KNOWLEDGE OF SERVANT'S INEXPERIENCE.

In order to hold a mine operator liable for injuries to a miner following a failure to warn and instruct the miner, there must be some evidence tending to show that the mine operator knew, either actually or constructively, that the miner was not experienced; and in order to hold an employer negligent on constructive knowledge of inexperience there must be something to suggest that warning and instruction were necessary.

Batesel *v.* American Zinc, Lead & Smelting Co. (Missouri Appeals), 176 Southwestern, 446, p. 448, May, 1915.

BLASTING—FAILURE TO GIVE WARNING.

In an action by a miner for damages caused by injuries received through the alleged negligence of a mine operator in blasting it is error as a matter of law for the court to instruct the jury trying the case that the failure of the operator to give warning that the blast was about to be fired constituted negligence on its part, as the question of negligence is a question of fact to be determined by the jury; and where all the evidence together shows that the blasts were fired before the miners came to work in the morning, or at noon, when they were away from work, or in the evening after they quit work, and that such blasts were always fired in the daytime because it was dangerous to work with dynamite by artificial light, and that warning was given to miners and employees who worked in the immediate vicinity, thus showing that the time selected for blasting was such that it was not reasonable to anticipate that miners would be in the vicinity; and where it also appears that the injured miner did not work at the place where the blasting was done, but only passed in going to and from his work, it can not, therefore, be said as a matter of law that the operator should have reasonably expected the miner to be within range of the explosion, and the entire question should be submitted to the jury for its determination.

American Bauxite Co. *v.* Dunn (Arkansas), 178 Southwestern, 934, p. 935, July, 1915.

OPERATOR DIRECTING EMPLOYEE TO WORK IN DANGEROUS PLACE.

A mine operator who directs a miner or employee to work in a trench in connection with the improvement and operation of the mining property and fails to take the proper steps to prop the bank of the trench to prevent caving, is guilty of negligence and liable for an injury to the employee if the conditions were such that an ordinarily prudent man would have taken steps to obviate the fault; and in such case the question of negligence is one of fact for a jury to determine.

Barnhart *v.* Waverly Brick & Coal Co. (Missouri Appeals), 176 Southwestern, 1108, p. 1110, May, 1915.

LAYING PIPE LINES ON SURFACE OF GROUND—LIABILITY FOR INJURY.

A person or corporation transporting natural gas in pipe lines laid on the ground in a public highway must exercise the highest degree of care to avoid injuries to those using the highway; and where an explosion was caused by a traction engine being driven over the pipe line and breaking it, and where the pipe line, covered with growing grass and weeds, was laid on the ground in the highway some 7 feet from a hedge fence, a court may refuse to admit evidence tending to show that other persons and corporations engaged in the transportation of natural gas did so by the means of pipe lines laid on the ground in public highways, as a person or corporation who maintains an exposed gas pipe through which natural gas is flowing on the surface of the ground within the limits of the public highway does an unlawful act and is liable in damages to one who without his fault breaks the pipe by driving a traction engine over it and is injured by an explosion of escaping gas.

Murphy *v.* Ludowici Gas & Oil Co. (Kansas), 150 Pacific, 581, p. 584, July, 1915.

EMPLOYMENT OF DISEASED CONVICT—LIABILITY FOR DEATH.

In an action for damages for the death of a convict employed by a mine operator, where it was averred in the complaint that the servants of the operator, within the line and within the scope of their duties, negligently put the intestate to work in mines while his feet were sore and the skin on his feet and ankles broken and punctured, and his feet thus exposed to dirt and cold and water, by reason of which and as a proximate result thereof he became sick and died, the plaintiff, in order to show the negligence charged as a proximate cause of the death of the convict, must show that the servants and agents of the operator knew of the condition of the convict's feet and ankles, or that they were guilty of negligence in not first ascertaining the condition of his feet and ankles, and where it appears that the condition of his feet and ankles was not open to ordinary observation. It was not the duty of the mine operator or of its servants and agents to make daily or frequent inspection of the convicts for the alleged ailments or troubles, and the operator and its agents had the right to assume that if the convict's feet and ankles were sore or swollen or pained him, he would have made the fact known to them.

Pratt Consolidated Coal Co. *v.* Bozeman (Alabama), 68 Southern, 887, May, 1915.

RIGHT OF INJURED RESCUER TO RECOVER.

A person who attempts to rescue another who has been put in peril by the negligence of a third person may maintain a cause of action against such third person for injuries sustained in attempting the rescue; but this right of action rests entirely upon the ground that the peril to which the person was exposed was caused by the negligence of such third person sought to be made liable in damages.

Taylor Coal Co. *v.* Porter (Kentucky), 175 Southwestern, 1014, p. 1017, May, 1915.

SCOPE OF EMPLOYMENT—MINER KILLED WHILE WORKING OUTSIDE OF EMPLOYMENT.

While the boss driver can not be presumed to have any right to order his assistant to take part in the work of repairing a wreck of trip cars, this being a distant branch of the mining service and outside of the scope of the boss driver or his assistant, yet a court may presume that the boss driver had authority to suspend his assistant from his regular employment and place him under the orders of a bank boss, a common superior, for any particular service for which he might be needed and as to which such bank boss might command or expect his service, and where such assistant boss driver was killed while aiding in such work outside of the scope of his employment the operator may be liable for his death, if it is shown that the bank boss ordered such assistant boss driver to perform the service he was rendering at the time he was killed, or if the bank boss impliedly authorized it by knowingly accepting the service, then the operator would be liable for the death of such assistant boss driver.

Republic Iron & Steel Co. *v.* Quinton (Alabama), 69 Southern, 604, p. 606, July, 1915.

INJURY TO SERVANT—THEORY OF CASE NOT CHANGED ON APPEAL.

Where a case was tried on the theory that the plaintiff, an injured miner, was an employee in the capacity of a servant of the operator and where the defendant procured the court to instruct the jury on the theory that the plaintiff was such servant, the operator can not, on appeal from a judgment for damages, insist that the injured miner was an independent contractor.

Clinton Mining Co. *v.* Bradford (Alabama), 69 Southern, 4, page 6, May, 1915.

ACTIONS FOR DAMAGES FROM ASSAULT AND BATTERY—GUARDS ACTING WITHIN SCOPE OF EMPLOYMENT.

In an action by a person injured by an assault made by agents, employees, and guards of a mining company, the mining company may be held responsible in damages if the guards and employees acted within the general scope of their authority, although they acted wantonly; and when the evidence and circumstances were open to conflicting inferences the question whether their acts were within the scope of the agents' authority is one of fact for the jury.

Pennsylvania Mining Co. *v.* Parnigan, 222 Federal, 889, p. 891, March, 1915.

LIABILITY FOR SLANDER UTTERED BY OFFICER.

A mining corporation is not liable for a slander uttered by an officer, though he is acting honestly for the benefit of the company and within the scope of his duties, unless the evidence shows that the corporation expressly ordered and directed the officer to say the particular words used, as a slander is the voluntary and tortious act of the speaker.

Republic Iron & Steel Co. *v.* Self (Alabama), 68 Southern, 328, April, 1915.

ACTIONS—PLEADING AND PROOF OF NEGLIGENCE.

PLEADING NEGLIGENCE—SUFFICIENCY OF COMPLAINT.

In an action for damages for the death of a miner, caused by the alleged negligence of a mine operator, the declaration is insufficient where there is no averment of any particular act or acts of omission to perform a duty required by law which, either singly or concurrently, can, within the established law upon the subject, be properly counted the proximate cause of the injury complained of, and where it is impossible to tell whether the liability, in the mind of the pleader, is of statutory or common-law origin, whether founded on defects inherently in the plan, construction, or equipment of the colliery, or one arising from wear and tear, or whether the case intended is one of improper supervision, and if the latter, whether the deficiency in that respect lay in the number, fitness, or inattention of the servants in charge, and the identity of the servants is not made to appear; and especially where the pleading proceeds upon the theory that the law requires the operator to give his miners an absolutely safe place in which to work and immediately to warn them of any impending danger, overlooking the fact that the requirement is reasonable, safety only to be secured by reasonable care under the circumstances.

Charnogursky *v.* Price-Pancoast Coal Co. (Pennsylvania), 94 Atlantic, 451, p. 452, March, 1915.

PLEADING—DEFECTIVE TRACKS—ALLEGATION SUFFICIENT TO SHOW NEGLIGENCE.

A complaint in an action by a miner for injuries which averred that the injuries were caused by a defect in the ways, works, machinery, or plant of the defendant operator, and averred that the track of a tram-car road used by the operator at or near where the plaintiff was injured was defective and that the defect arose from or had not been remedied owing to the negligence of the operator or of some person in the service or employment of the operator and intrusted by him with the duty of seeing that the ways, works, machinery, or plant were in proper condition is sufficient to show to a common intent that the gravamen of the pleading is the negligence of the operator or his servants to whom has been committed the duty of maintaining such tram track on which the plaintiff was working and limits the proof to the question of negligence with reference to maintaining the tram track and sufficiently states a cause of action.

Birmingham Fuel Co. *v.* Stocks (Alabama), 68 Southern, 568, May, 1915.

ACTION AGAINST CORPORATION FOR PERSONAL INJURIES—VENUE.

Section 6112 of the Code of Alabama authorizes all suits against mining corporations or corporations generally in any county where they do business by an agent, except as to actions for personal injuries, and these must be brought in the county in which the injury occurred or in the county in which the plaintiff resides, if the offending corporation does business by an agent in the county of the plaintiff's residence; and this section authorizes an action by an injured miner to be brought in the county where the injury occurred, whether the corporation did business other than by an agent or not.

American Coal Corporation *v.* Roux (Alabama), 68 Southern, 970, May, 1915.

NEGLIGENCE OF OPERATOR OR INDEPENDENT CONTRACTOR—QUESTION OF FACT.

In an action against a coal-mine operator for the death of a miner caused by the fall of rock from the roof of the mine, it becomes a question of fact to be determined by the jury trying the case whether the proximate cause of the miner's death was due to the negligence of the mine operator or whether the mine was operated by an independent contractor and for whose negligence the mine operator was not responsible; and where the evidence shows a written lease from the mine owner by which the mine was leased to a third person named, to be operated by him, but where the evidence shows also that the mine operator issued checks to the miners in its own name in payment for labor and that such checks were good in the purchase of merchandise at the commissaries of the mine operator and that the mine operator deducted from the miners' wages certain insurance premiums, as shown by the records of the timekeeper of the mine operator, and that the checks for time or wages were turned in at the office of the mine operator and the money was paid thereon by the operator, and that the mine was in fact owned by the defendant operator, and that the mules and cars used for hauling coal belonged to the operator and that the alleged lessee had before that time been employed by and worked for the mine operator, though at the time of the alleged injury he had an office next to the office of the mine operator where the time of the miners was turned in and kept, and that the miners went directly from such office to that of the mine operator and got checks in payment for time employed or labor performed, it was error for the court to charge the jury that such lessee was an independent contractor.

Amerson *v.* Corona Coal & Iron Co. (Alabama), 69 Southern, 601, June, 1915.

COMPLAINT FOR DAMAGES FOR ASSAULT—ADMISSIBILITY OF EVIDENCE.

In an action by a union leader and organizer against a mining company for damages for injuries caused by an assault by the defendant's employees where the complaint charged that the defendant stationed at his mine certain named persons, agents, and employees for the purpose of guarding and protecting the property, and while such agents and employees were acting within the general scope of their employment, assisted by and under the personal guidance and direction of the defendant superintendent, they waylaid the plaintiff while on the public highway and with deadly weapons unlawfully, wantonly, wickedly, and maliciously beat, bruised, and wounded him, an objection to the introduction of evidence on the ground that the complaint failed to state a cause of action is unavailing as against such an attack, every reasonable intendment will be indulged.

Pennsylvania Mining Co. *v.* Jarnigan, 222 Federal, 889, p. 890, March, 1915.

MAINTAINING DANGEROUS PLACE.

DUTY TO FURNISH SAFE PLACE—NONDELEGABLE DUTY.

The duty of a master or mine operator to provide a safe working place and machinery for his employees can not be delegated so as to absolve him from liability in case of failure of a vice principal to perform such duty.

Mace *v.* Carolina Mineral Co. (North Carolina), 85 Southeastern, 152, p. 154, May, 1915.

SAFE PLACE AND APPLIANCES FURNISHED—PERSONAL SUPERINTENDENCE NOT REQUIRED.

A mine operator who furnishes a safe place, proper machinery and appliances, and competent men to perform the work, is not required to personally superintend all the details of the work and can not be held liable for every negligent act of his employees in the use of such machinery and appliances.

Lathi *v.* Tamarack Mining Co. (Michigan), 152 Northwestern, 907, p. 910, June, 1915.

OPEN SHAFT—DUTY AND LIABILITY.

A coal company is not guilty of negligence, in the absence of a statute, in failing to have a shaft of an abandoned mine filled up or covered where it was sufficiently inclosed to prevent persons from falling into it. Section 4359 of the Kentucky statutes providing that wells and pits sunk for salt water, or for any other purpose, when abandoned or not in use, shall be filled up or inclosed, applies only to salt or saltpeter works, as shown by the title and body of the act, and does not apply to shafts in abandoned coal mines.

Taylor Coal Co. *v.* Porter (Kentucky), 175 Southwestern, 1014, p. 1016, May, 1915.

SAFETY OF PLACE A QUESTION OF FACT.

Where it is made to appear that a rock which fell from the roof and injured a miner was loose and dangerous when he went to the entry or station to go to work, and was not in fact loosened by the very work which the miner was doing, then the miner would be entitled to recover for the alleged injury; but if the miner either by a shot or otherwise made the rock loose and dangerous by the work which he did, then the mine operator would not be liable for the resulting injury; but neither of these conditions is a question of law for a court to determine, but is one of the fact for the jury on the trial of the case.

Zelavin *v.* Tonopah Belmont Development Co. (Nevada), 149 Pacific, 188, p. 190, June, 1915.

DEATH OF RESCUER—LIABILITY OF MINE OPERATOR.

The fact that a coal company knew, or in the exercise of ordinary care could have known, that black damp had accumulated in a shaft into an abandoned mine is not sufficient to establish negligence and render the company liable for the death of a person occasioned by an accumulation of such black damp and poisonous gas in the shaft, where the person losing his life voluntarily entered and went down the shaft for the purpose of rescuing a fellow workman who had gone into the shaft and had been overcome by the poisonous gas, as the coal company could not have anticipated that the accident in which such person lost his life would have happened in the manner it did, and as the character of the accident was such that no amount of care short of filling up or cleaning the shaft could have guarded against it, and the coal company, so far as the evidence showed, was under no duty to do either.

Taylor Coal Co. *v.* Porter (Kentucky), 175 Southwestern, 1014, p. 1016, May, 1915.

DEFECTIVE APPLIANCES.

FAILURE TO INSPECT—PROXIMATE CAUSE.

In an action by a miner for injuries caused by a defective skip or improper operation of a skip, and where there was an averment in the declaration to the effect that it was the duty of the mine operator to provide and maintain a proper and suitable signaling apparatus, including a reasonably safe signal-bell line and bell, for the purpose of notifying and conveying signals from the men underground to the engineer, and alleging a negligent failure to furnish and maintain such suitable signaling apparatus, and negligently failing to inspect and repair the same, these are sufficient allegations to admit testimony relative to the inspection of the signaling apparatus by the mine operator. But conceding that the signaling apparatus should have been inspected from time to time and conceding a failure to so inspect, yet in the absence of testimony tending to prove that it was not in working order, or that the failure to so inspect caused or contributed to the injury, and where, on the other hand, the testimony shows the signaling service to have been in working order and that the skip was operated in obedience to signals, the plaintiff under such allegations in his declaration is not entitled to recover.

Paskovan *v.* Allouez Mining Co. (Michigan), 153 Northwestern, 684, p. 685, July, 1915.

USING DEFECTIVE SKIP.

A mine operator can not escape liability for damages for the death of a miner caused by the use of a skip while carrying miners out of a mine on the ground that a rule existed prohibiting miners from riding out of the mine on the skip, where the testimony of the employees is to the effect that they knew nothing of the existence of a rule forbidding them to use the skip and where the mine owner himself and his foreman set the example and rode in the skip several times, using the same signals which had been used for a bucket previously in use and where it also appears that the skip could easily have been made safe and thereby prevented the accident.

Hardister *v.* Richardson (North Carolina), 85 Southeastern, 304, p. 305, May, 1915.

FAILURE TO DISCOVER DEFECTS IN TRAM TRACKS—LIABILITY.

In an action by a miner for injuries caused by tramcars, and because of a defect in the tram track that rendered its use hazardous, proof of notice of such defect to the mine operator is not essential to liability, as it is the duty of the operator to discover and remedy such defects, and negligent failure to discover is as potent in sustaining the plaintiff's case as negligent failure to remedy, and either is sufficient to sustain a cause of action by a miner for injuries caused by tramcar on such defective track.

Birmingham Fuel Co. *v.* Stocks (Alabama), 68 Southern, 568, May, 1915.

DEFECTIVE LINK—FAILURE TO INSPECT.

In an action by a miner for personal injuries caused by a broken link in a chain and where there are certain signs on the exterior of the link indicating imperfect welding and the real fault would have been fully exposed by a due inspection and test before the accident, the fault or defect is a structural defect of which the employer is presumed to have knowledge, and if the defect shows exterior signs which render it ascertainable by reasonable inspection it is to all intents and purposes patent and the rule applicable to that class of defects governs.

Case *v.* Lehigh Valley Coal & Navigation Co. (Pennsylvania), 94 Atlantic, 252, p. 253, March, 1915.

FAILING TO EQUIP CARS WITH AUTOMATIC COUPLERS.

A mine owner and smelter operator using locomotives and cars running on tracks can not avoid a charge of negligence in failing to use automatic couplers, because such locomotives and cars are operated entirely in his own yards and by him as a private carrier, or because of the fact that its locomotives and cars were smaller and the track narrower than standard gage, as it is in all essential respects a railroad.

Consolidated Kansas City Smelting & Refining Co. *v.* Schulte (Texas Civil Appeals), 176 Southwestern, 94, p. 95, May, 1915.

LIABILITY FOR NEGLIGENCE OF FELLOW SERVANT.

NEGLIGENCE OF TRIP DRIVER—QUESTION OF FACT.

Where a track repairer in a mine was directed to get his tools, put them on a trip, and bring them to a certain designated place, and while so doing and while riding on the bumpers of the rear car was injured by reason of the sudden stopping of the trip traveling at a high rate of speed, and the car thereby caused to kick up so suddenly that his head came in contact with the roof of the mine, and was thereby injured, the question of the negligence of the trip driver in running the cars at too great a rate of speed and in suddenly stopping the motor, causing the car to kick up, is to be submitted to and tried by a jury, and a court will approve a finding of a jury on the question of negligence under such circumstances.

St. Bernard Mining Co. *v.* Ashby (Kentucky), 175 Southwestern, 626, April, 1915.

EMPLOYEES MAKING THEIR WORKING PLACE AND APPLIANCES.

Where an oil-drilling company did not undertake the duty of furnishing such a structure as a tubing board, but merely supplied suitable materials and reasonably competent fellow servants by which a tubing board could be constructed and reasonably adapted to the work, and where the duty of installing such tubing board as an appliance for use was intrusted or assumed by the employees themselves, the employer is not answerable to one of such employees for the negligence of himself or of a fellow employee in the construction of such tubing board; but if the employee had been sent upon a tubing board already fixed and placed in the derrick, or if he had been sent to use a tubing board in his work which the oil company undertook to install, or where the oil company undertook either in person or through another employee to supervise the installation, and an injury resulted, the rule would be otherwise.

Barnsdall Oil Co. *v.* Ohler (Oklahoma), 150 Pacific, 98, p. 100, June, 1915.

MINERS MAKING THEIR OWN APPLIANCES.

Where an oil company provided an adequate and readily accessible stock of appliances in good condition from which to make a selection, and the imperfection of an instrumentality selected therefrom was or ought to have been apparent to the employee who selected it, the oil company can not be held responsible for injuries which were caused by the instrumentality, whether the injured person was himself the employee who made the selection or whether it was done by a coemployee.

Barnsdall Oil Co. *v.* Ohler (Oklahoma), 150 Pacific, 98, p. 101, June, 1915.

FAILURE OF TRIP DRIVER TO LIGHT CARS—INJURY TO MINER IN HAULAGEWAY.

A crosscut in a mine was used as a haulageway for hauling ore from the working places in the mine to the foot of the shaft and also for a passageway for the miners going from the shaft to their place of work and was unlighted except by miners' lamps. The cars were drawn by a cable which ran over a series of rollers at stated distances along the track and also over or around a wheel at the base of the shaft, and the loaded cars when drawn by this cable traveled ordinarily at a speed somewhat faster than a man walks. The conductor, the man accompanying each trip of cars, was required to display a light as a warning to the miners of the approach of the cars, and it was customary for the conductor to have a light on the front end of the front car as a signal to miners using the passage. While this crosscut was straight, so that a light could be seen through its entire distance, yet a miner passing from the shaft into the crosscut could ascertain whether or not cars were running by looking at the wheel around or over which the cable ran and could tell the direction in which the cars were running by feeling the rope back of the wheel or by putting his foot on it, as well as by watching the light. The light used as a signal was not permanently attached to any car, but there was a little hole on the end of the car to hook on the lamp, and it was the duty of the conductor accompanying the trip to attach the light to the car by placing the hook on the lamp in the hole; the conductor also carried a lamp of his own on his hat. On a particular trip the lamp placed by a conductor on the front end of the head car was broken at or near the point where he started with the loaded trip, and he ran the cars into the crosscut without renewing the light at the front end of the car. During this time two miners entered the crosscut from the base of the shaft, one of them feeling the rope with his hand and the other with his foot, and discovered that it was not moving and proceeded through the crosscut, and while so doing were met and injured by the trip of loaded cars without any light or signal on the front car and with no light other than that on the hat of the conductor, who at the time was on the rear end of the second car with his head or hat but slightly above the top of the car. Under such circumstances and this state of facts the negligence of the conductor in failing to renew the light or in failing to place a light on the front end of the head car was the negligence of a fellow servant and for which the mine operator was not liable to the injured miner.

Lahti *v.* Tamarack Mining Co. (Michigan), 152 Northwestern, 907, p. 908, June, 1915.
See Kaukola *v.* Oliver Mining Co., 159 Michigan, 689; 124 Northwestern, 591.

FAILURE OF MINERS TO BLAST ROCK FOR TRAMMER—POWER TO GIVE ASSURANCES OF SAFETY.

A mine operator is not liable for the death of a trammer in a stope in the process of development by miners who excavated the ore and passed it down from openings in the stope to the level where it was loaded on the cars by the trammers, and where the deceased trammer and others working with him requested the miners to blast a large rock discovered by them and which the miners promised to do "in about 15 minutes," but before the time had elapsed the rock fell and killed the miner, as the miners under the circumstances shown were fellow servants of the trammers and the duty of the miners among others was to blast such rocks when requested to do so by the trammers, and the blasting of such rocks was done by the miners for the safety of the trammers, because they were not possessed of sufficient ability or training to use explosives; and it can not be said that the mine operator undertook to protect the trammers against injury from such large rocks, and especially where the particular rock had remained in the same position for four days, and where the danger of working below the rock was apparent and readily comprehended by the trammers, and the neglect of the miners, if it can be called neglect, not to blast instantly, when requested by the trammers to do so, can not be charged to the operator, nor can it be said to have been the proximate cause of the trammer's death; and if the miners under such circumstances gave any statement of assurances of safety to the trammers, it can not be deemed to have been given in a representative capacity and in fact can only be given by a person representing the master, such as a vice principal.

Lesh *v.* Tamarack Mining Co. (Michigan), 152 Northwestern, 1021, p. 1024, June, 1915.

MINER'S WORKING PLACE—SAFE PLACE.

SAFE-PLACE DOCTRINE—MINER'S WORKING PLACE.

Where the place to be furnished by a mine operator is not furnished as in any sense a permanent place of work, but is a place in which surrounding conditions are constantly changing, and instead of being a place furnished by the master or mine operator for employees within the above rule, it is a place the furnishing and preparation of which is in itself a part of the work which the miners are employed to perform, then the rule as to the duty to furnish a safe place does not apply, but the duty of the master or mine operator is performed by using reasonable care or furnishing suitable material and employing capable and efficient men to do the work.

Mace *v.* Carolina Mineral Co. (North Carolina), 85 Southeastern, 152, p. 154, May, 1915 .

See Consolidated Coal & Mining Co. *v.* Floyd, 51 Ohio St. 542, Northeastern, 610, 25 L. R. A. 848.

Petaja *v.* A. J. Mining Co., 106 Michigan, 463, 64 Northwestern, 335, 58 Am. St. 505, 32 L. R. A. 435.

SAFE PLACE—MINER'S DUTY TO MAKE PLACE SAFE.

Where a miner or servant is employed in performing labor which necessarily changes the character of the place for safety where the work is being done as the work progresses, and consequently is likely to become dangerous at any moment, the miner assumes the risk incident to such work, and the general rule that a master must furnish his servant a reasonably safe place in which to work, does not apply.

Zelavin *v.* Tonopah Belmont Development Co. (Colorado), 149 Pacific, 188, p. 189, June, 1915.

MINER MAKING PLACE SAFE—DUTY TO REMOVE ROOF.

A miner injured by slate falling from the roof of the room in which he was at work can not recover from the operator for injuries received where it was his duty to sound the roof and take down the slate if he found it loose, and where it is shown that he did sound the roof and found it loose, but failed to take down the slate that fell and caused the injury.

Peerless Coal Co. *v.* Copenhaver (Kentucky), 176 Southwestern, 1002, p. 1003, June, 1915.

INJURY FROM FALL OF ROOF—DUTY OF INSPECTION.

Where the respective duties of a miner and the mine operator in regard to roof inspection were governed by unwritten rules of the operator, and where there is a sharp conflict in the evidence as to what such rules were, an injured miner contending in an action for damages for an injury caused by the fall of the roof, that after the room neck was excavated and the operator placed its tracks for hauling therein, the operator assumed the duty of inspecting and keeping such room neck in a reasonably safe condition so that rock would not fall from the roof; and where other miners employed in the same mine at the same time testified that it was not the duty of the operator to inspect the room necks, but that a miner working in a room was required to inspect the neck of his room the same as the body, admitting that it was the duty of the operator to repair room necks when notified by miners that they were dangerous, and where other miners testified that it was the custom and duty of the operator to inspect the room necks each morning before the miners began work to ascertain if there was in the roof thereof any loose rock, a court may under such circumstances properly refuse to direct a verdict for the defendant operator, as the weight of the evidence on the controverted point is properly for the jury, and the court on appeal must be bound by its determination.

Matthews *v.* Central Coal & Coke Co. (Missouri), 177 Southwestern, 650, p. 651, June, 1915.

MINER DRIVING ENTRY AND MAKING HIS WORKING PLACE.

The duty resting upon a mine operator to furnish safe ways, works, and machinery does not apply, and the operator is not liable for an injury to its employee, where a mine operator employs an independent contractor who is an experienced miner to drive a heading in the mine and put down tracks for the operation of the mine, under the rule that where the instrumentality causing the injury was still incomplete at the time of the injury and the injured miner was engaged in the work of bringing it to completion and where the unsafe condition from which the injury arose was incidental to the work thus undertaken by him.

Woodward Iron Co. *v.* Wade (Alabama), 68 Southern, 1008, p. 1010, June, 1915.

CONTRIBUTORY NEGLIGENCE OF MINER.

CONTRIBUTORY NEGLIGENCE DEFINED.

Contributory negligence in its legal significance is such an act or omission on the part of the person injured, amounting to a want of ordinary care, as concurrent or cooperating with some negligent act or omission of another person, approximately causes or contributes to cause the injury complained of.

Consolidated Kansas City Smelting & Refining Co. *v.* Schulte (Texas Civil Appeals), 176 Southwestern, 94, p. 95, May, 1915.

CONTRIBUTORY NEGLIGENCE DEFEATS RECOVERY.

In an action for damages for injuries received by a miner the miner can not recover if he himself was guilty of negligence which proximately contributed to the injury, as such contributory negligence is a complete defense to such an action.

Birmingham Fuel Co. *v.* Stocks (Alabama), 68 Southern, 568, p. 569, May, 1915.

RECOVERY NOTWITHSTANDING CONTRIBUTORY NEGLIGENCE.

A miner injured by an explosion of fire damp or gas, the presence and extent of which were not properly noted but only guessed at by the fire inspector, notwithstanding the fact that he was guilty of contributory negligence in fanning or blowing the gas toward an open and lighted lamp, may recover damages for such injuries.

Oplotnik *v.* Cherokee & Pittsburg Coal & Mining Co. (Kansas), 148 Pacific, 616, May, 1915.

MINER'S KNOWLEDGE OF DANGER—CONTRIBUTORY NEGLIGENCE.

An action against a coal-mine operator for the death of a miner caused by being caught between a loaded coal car and a prop near the track, and caused presumably by the miner being caught between the car and the prop by attempting to sprag the car to prevent its running too fast down the track, can not be maintained where the miner had knowledge of the position of the props, had passed them many times, and where he had in fact assisted in placing the props, and where it appeared that there was another and safer way to prevent the car from running too fast.

Bon Jellico Coal Co. v. Walker (Kentucky), 175 Southwestern, 629, p. 630, April, 1915.

DANGER IMMINENT.

Where an injury to a miner arose out of the negligence of a mine operator or master, yet if the danger from such negligence was so great that the miner or servant could not hope to escape injury by the exercise of reasonable care, then he will be guilty of contributory negligence and can not recover, notwithstanding the operator's negligence.

Peerless Coal Co. v. Copenhaver (Kentucky), 176 Southwestern, 1002, p. 1110, June, 1915.

KNOWLEDGE OF DANGER—MINER CREATING DANGEROUS CONDITION.

In an action by a miner for damages for injuries caused by slate falling from the roof of the room in which he was working and where the evidence was conflicting as to whether it was the duty of the miner to prop the roof at the place of the injury or to sound the roof and take down the slate if he found it loose, it was held to be error for the trial court to refuse at the request of the defendant the following instruction:

> If you believe from the evidence plaintiff removed the coal from under the slate which afterwards fell on and injured his leg, thereby taking from under said slate its natural support, and thereafter, and before said slate so fell, he tested it and ascertained that it was loose, and you also believe from the evidence that it was the duty of the plaintiff, under the contract he had with defendant company, to take down such loose slate, but that he went under said slate, and while under it it fell on and injured him, you ought to find for defendant company.

The reason given is that such an instruction was necessary to present the defendant's side of the case, where without such an instruction the jury might conclude that the facts relied upon did not constitute contributory negligence or assumed risk, and where the defense relied upon in the instruction was actually pleaded and sustained by the proof and where the trial court gave no instructions presenting to the jury the duty of the miner with reference to the overhanging slate when the conditions were such as to make it impractical to prop the roof.

Peerless Coal Co. v. Copenhaver (Kentucky), 176 Southwestern, 1002, p. 1003, June, 1915.

MINER PLACING HIMSELF IN DANGEROUS POSITION.

A mine operator is not liable for the death of an experienced miner employed as a foreman because of his experience, where the work he was to do on the day of and at the time of the accident, was left, in respect to the method and manner of doing it, to his own judgment, and where he was perfectly free to exercise his own common sense and skill in the mining operation, and where, from the description of the condition immediately before he was killed, he must have known that by digging under a projecting or overhanging bank of dirt or rock he was placing himself in a dangerous position, as the unsupported bank would necessarily cave in, and a man of ordinary sense and of prudence would know of such dangers and appreciate the risk of cutting out the foundation upon which a bank of dirt rests and leaving it overhanging without any support, brace, or prop to prevent its falling in and crushing him; and the danger of such a place was so imminent that any ordinarily prudent man would not have so cut underneath the bank as to weaken its support and cause it to fall, or if this was necessary to be done, he would have taken measures to brace it in some way as the work progressed.

Mace *v.* Carolina Mineral Co. (North Carolina), 85 Southeastern, 152, p. 153, May, 1915.

KNOWLEDGE OF DANGER—MINER'S OPTION AS TO METHOD OF WORK.

Where the danger is obvious and is of such a nature that it can be appreciated and understood by the miner as well as by the operator, or by anyone else, and where the miner has as good an opportunity as the operator or anyone else of seeing what the danger is and is permitted to do his work in his own way and can avoid the danger by the exercise of reasonable care, the operator is not liable for injuries received by the miner in consequence of the condition of things which constitute the danger, and the resulting injury must be regarded as due to his own want of care.

Mace *v.* Carolina Mineral Co. (North Carolina), 85 Southeastern, 152, p. 154, May, 1915.

DRIVING TRACTION ENGINE OVER PIPE LINE LAID ON SURFACE.

A person driving a traction engine on a highway has the right to assume that the highway is safe from one side to the other, and is under no obligation to make an examination for the purpose of ascertaining whether or not the gas pipes were laid thereon; and it is not contributory negligence for the driver to fail to look for hidden pipe lines, although in a community where natural-gas pipe lines are known to be laid in the public highways; and the driver of the engine would not be subject to the charge of contributory negligence even if he had knowledge of the presence of the pipe line, if he had exercised care and adapted his conduct to the existing condition and employed such care as may justly be regarded as ordinary, in view of the knowledge of the existence of the obstruction.

Murphy *v.* Ludowici Gas & Oil Co. (Kansas), 150 Pacific, 581, p. 585, July, 1915.

EFFECT OF INTOXICATION.

The mere fact that a miner was intoxicated at the time he received an injury does not of itself show such contributory negligence as will defeat his recovery in damages for the injury, but it is a circumstance to be considered in determining whether or not his intoxication contributed to the injury.

American Bauxite Co. *v.* Dunn (Arkansas), 178 Southwestern, 934, p. 935, July, 1915.

ASSUMPTION OF RISK.

RISKS ASSUMED.

ASSUMPTION AS TO EMPLOYEE'S COMPETENCY.

A master or mine operator may assume that an adult person in accepting or engaging employment is competent to perform the particular work in which he engages, and that he assumes the dangers ordinarily incident to the carrying on of the work.

Batesel *v.* American Zinc, Lead & Smelting Co. (Missouri Appeals), 176 Southwestern, 446, p. 448, May, 1915.

RISKS OF EMPLOYMENT—MINER'S KNOWLEDGE OF MINING.

It is necessary in mining operations for mine operators to employ skilled miners, and such miners are frequently better informed of the risks and necessities of the particular branch of the mining than the employer himself; and to hold that such a miner with full knowledge of the dangers and attendant risks could rely upon his own judgment as to the liability of a given slab or bowlder to fall, and then, after an injury, hold the mine operator liable for the resulting injury, would be to make the operator responsible for the lack of judgment of the miner. In other trades and callings it is elemental law that if an employee possesses equal or superior information to the employer in regard to the danger from a given place or appliance, then in case of injury the employee is held in law to have assumed the risk as an incident to his employment, and this rule of law applies to injuries received by experienced miners resulting from falls or slabs and bowlders in mines, as well as other injuries from different causes in mining operations.

Woodward Iron Co. *v.*. Wade (Alabama), 68 Southern, 1008, p. 1011, June, 1915.

KNOWLEDGE OF PHYSICAL INFIRMITIES—INJURY TO EPILEPTIC.

An epileptic who is subject to fits and who is injured by falling into a dangerous place when stricken with a fit can not recover for the injury without proving that he was subject to such fits and that he himself did not know it and that the employer did know it, and knew, or had cause to know, that the plaintiff did not know his condition; but if the plaintiff knew that he was subject to fits and that such a fit might cause him to fall into a dangerous place, then he can not recover. An employee laboring under any physical disability of which he has full knowledge, who accepts employment with another, can not recover damages of the employer in the event he sustains personal injuries during the employment, where such injuries were directly caused by his physical disability, and the fact that the employer directed the plaintiff to work in a dangerous place will not create a liability under such circumstances, as persons not under contractual disability should be and are permitted to determine for themselves whether their physical infirmities, known to themselves, shall debar them from any particular employment, and such persons must estimate for themselves the chances of injury, and it would be unjust as well as illogical to visit the consequence of their choice upon their employers; it may be a matter of delicacy as well as difficulty for an employer to undertake in each case to determine the nature and extent of the malady or infirmity of one who offers to work, or the likelihood of injury resulting therefrom, and an employer may, without legal liability, accept the judgment of a person offering to perform services who knows his own condition, and under such circumstances the employee assumes the risk of injury when he undertakes to do the work for which he was employed.

Tennessee Coal, Iron & Railroad Co. *v.* Moody (Alabama), 68 Southern, 274, p. 275, April, 1915.

SHOVELER FIRING POP SHOTS.

A person employed in a mine as a shoveler is supposed to know that bowlders are to be broken up by the use of dynamite fuses and caps and that men are specially employed to perform such work, and he must therefore know that this is a special employment outside of what he contemplates when he goes into a mine to work as a shoveler, and that there is a certain skill or experience necessary, and that the exploding of the dynamite is necessarily accompanied with certain risks and dangers; but if as a shoveler he had been informed that shovelers in a particular mine must also engage in the work of shot firing and he accepted the employment, the duty of the operator or employer would be satisfied, because by knowing that he would be required to handle and explode dynamite as a shot firer, and after becoming aware of such fact he accepted the employment and engaged in the work of shoveling and shot firing, he would thus have held himself out as experienced and able to perform the work, and the master would not have been required to give warning or instruction; and when the injured miner did go into the mine to work and on the first night, while there, discovered that the shovelers fired their own shots, he realized what his duties under his employment would require him to do, and the shoveler thus had constructive knowledge of what was required of him, and being an adult of ordinary intelligence he must be held to have assumed the risk incident to the undertaking.

Batesel *v.* American Zinc, Lead & Smelting Co. (Missouri Appeals), 176 Southwestern, 446, p. 449, May, 1915.

KNOWLEDGE OF DANGER—RESCUE OF ANOTHER FROM PERIL.

A mine operator who is himself free from negligence can not be made liable in damages on account of injuries sustained by a person who, at his request, comes to the assistance of one who is in danger or exposed to peril; but in order to render the person making the request liable for damages he must by his own negligence have produced the peril. In the absence of such negligence and in the presence of the peril the rescuer, whether acting voluntarily or on request, assumes the risk incident to the attempted rescue.

Taylor Coal Co. *v.* Porter (Kentucky), 175 Southwestern, 1014, p. 1017, May, 1915.

KNOWLEDGE OF PROPS DANGEROUSLY NEAR A TRACK.

A miner who assists in placing props supporting the roof near the track over which the coal is hauled out of his entry and who has many times passed such props and who well knew the dangerous situation and the danger in attempting to pass between a loaded car and the props, assumes the risk incident to the undertaking and there can be no recovery for his death caused by being caught between a loaded car and a prop, presumably in attempting to sprag the wheel to prevent the car from running too fast down the track, when there was another and safer way to prevent the car running down the incline.

Bon Jellico Coal Co. *v.* Walker (Kentucky), 175 Southwestern, 629, p. 630, April, 1915.

RISKS NOT ASSUMED.

OPERATOR'S NEGLIGENCE.

A miner never assumes a risk arising from the operator's negligence.

Matthews *v.* Central Coal & Coke Co. (Missouri), 177 Southwestern, 650, p. 653, June, 1915.

DANGER NOT IMMINENT.

Where an injury arises out of the negligence of a mine operator or a master, then the mere knowledge of the condition on the part of the miner or servant will not bar him from recovery on the ground of assumption of risk; but if the danger is not glaring and imminent then even if the miner or servant has knowledge of the situation he does not assume the risk arising from the operator's negligence.

Barnhart *v.* Waverly Brick & Coal Co. (Missouri Appeals), 176 Southwestern, 1008, p. 1110, May, 1915.

FAILURE TO EQUIP CARS WITH AUTOMATIC COUPLERS.

A miner employed by a smelter company does not assume the risk of the negligent failure of the company to equip its cars with automatic couplers; and the failure to equip its cars with automatic couplers is a defect within the meaning of section 6645 of the Revised Statutes of Texas.

Consolidated Kansas City Smelting & Refining Co. *v.* Schulte (Texas Civil Appeals), 176 Southwestern, 94, p. 96, May, 1915.

MINER WORKING UNDER DIRECTIONS OF FOREMAN—ASSURANCES OF SAFETY.

A miner or employee working in a trench at the direction of a mine foreman is not to be charged with the assumption of risk as a matter of law from the mere fact that he made no complaint and received no express or affirmative assurance from the operator that the place was safe, and where he testified that he was watchful of danger or that there was none; and in such case he had a right to rely on the superior judgment of the foreman and the presence and direction of such foreman to go into the trench and work were equivalent to an assurance on the foreman's part that the employee could safely proceed. The operator under such circumstances can not escape liability on the ground that the danger was as well known to the employee as to the foreman, and while the employee could see the conditions present as well as the foreman, there is nothing that can justify a court in saying that he was well aware of the danger where the employee had not dug in the trench and was not an experienced trench digger; but in such case the foreman is presumed to have a superior knowledge and his position and authority imply this.

Barnhart *v.* Waverly Brick & Coal Co. (Missouri Appeals), 176 Southwestern, 1108, p. 1110, May, 1915.

USE OF EXPLOSIVES.

INJURY FROM UNEXPLODED BLAST.

A certified miner who kept his explosives in a box with his tools in the mine is not entitled to recover for injuries caused by an explosion where he drilled a hole in a seam of coal and inserted a stick of dynamite with a detonating cap attached properly covered and tamped with a squib attached in a blasting barrel for the purpose of firing the charge, and where on a particular occasion the charge did not explode because the powder used in connection with the dynamite was damp and moist, and where on the following day the miner with a pick attempted to break up a large lump of coal blown down by a subsequent blast, and where while striking the lump of coal with the pick an explosion occurred causing the injuries complained of, as under such circumstances the mine owner had no greater reason to anticipate that water dripping on the miner's box wet the powder, and he should not therefore be held liable for a consequence not fairly to be expected.

Lehigh Valley Coal Co. *v.* Calcausky, 222 Federal, 664, April, 1915.

USE OF DANGEROUS EXPLOSIVES.

Where a mine operator furnished for use a more powerful and sensitive explosive which was not suitable to the accomplishment of the work which a miner was employed to do, thereby creating abnormal risks to which the miner became exposed without giving him notice of the fact so that he could guard himself against them, the mine operator may be liable for resulting injuries; but if the operator gave the miner notice of the change of explosives, or if he had knowledge of it, obtained in any manner, then the miner assumes the risk incident to it and can not recover of the operator for injuries resulting. And in an action by a miner for injuries caused by improper explosives, a general verdict in favor of the plaintiff can not stand as against an answer by the jury to a special interrogatory in which it is expressly found that the miner had been warned by the operator and that he knew of the stronger explosive, and that the use of such explosive under the particular circumstances and for the purpose required was proper.

Johnson *v.* Butte Elex Scott Copper Co. (Montana), 149 Pacific, 717, p. 719, June, 1915.

INJURY TO TRESPASSERS—LIABILITY.

CARE REQUIRED TOWARD TRESPASSERS.

The right of dominion and use of property can not be made a shield or cover for malicious acts on the part of the owner, but owners owe no duty to strangers on their lands without right except abstention from intentional injury, and such strangers are classified as trespassers and bare licensees and can not recover for injuries caused by excavations, defects in the premises, contact with running machinery, or the like, as the proprietor owes them no active duty and so long as he does them no intentional injury he is not liable for anything that may befall them; but in order to create a liability for injuries in favor of one who comes upon the premises of another on business, in some sense of the term, there must be an invitation to come upon some matter or occasion of material interest or benefit to the owner, and not a mere curiosity or other considerations exclusively personal to such trespasser or licensee.

Dickinson *v.* New River & Pocahontas Consolidated Coal Co. (West Virginia), 85 Southeastern, 71, p. 72, April, 1915.

DUTY TO TRESPASSING CHILDREN.

A coal-mine operator ordinarily is under no duty, in the handling of its coal cars and motors on its private railway between the mouth of its mine and its yards or tipple, to keep a lookout for children of employees on its tracks, although they reside in houses of the corporation on its premises and near its tracks.

Dickinson *v.* New River & Pocahontas Consolidated Coal Co. (West Virginia), 85 Southeastern, 71, p. 72, April, 1915.

METHODS OF OPERATING.

FREEDOM IN THE USE OF PROPERTY—LIMITATIONS.

Out of the dominion and use of private property incident to the ownership thereof, limited only by the right of the property of others to freedom from injury from such use, springs the principle of immunity from liability for injuries to trespassers and bare licensees and only upon the highest and most imperious considerations is this right of dominion and use limited, and it is with great caution that courts and legislatures impose restraints upon the use of property and none are ever laid thereon without careful definition of their extent.

Dickinson *v.* New River & Pocahontas Consolidated Coal Co. (West Virginia), 85 Southeastern, 71, p. 72, April, 1915.

RESTRAINTS IN USE OF PROPERTY—APPLICATION TO PERSONS.

A legislature may, under the police power of the State, impose limited restraints upon the use of private property, in the form of regulations thereof, and it may authorize municipal corporations to do the same; but the Constitution withholds from the legislature power to deprive a citizen of his property or its use, except for public purposes, and only on payment of just compensation; and the common law imposes an obligation upon a property owner to use it in such manner as not to injure that of another; but this obligation is in favor of other property and not in favor of persons disassociated from the property, and a stranger's right under this principle pertains to his property and the enjoyment and use thereof and not to his person.

Dickinson *v.* New River & Pocahontas Consolidated Coal Co. (West Virginia), 85 Southeastern, 71, p. 72, April, 1915.

RIGHTS OF OWNER OF COAL TO MINE AS AGAINST SURFACE OWNER.

A mine operator having the exclusive right to mine coal upon or under a certain described tract of land has the right of possession even as against the owner of the soil, so far as is reasonably necessary to carry on his mining operations, and has, as grantee of the minerals, the right to dig and carry away so much of them as he can excavate from the soil without injury to the surface owned by the grantor, the mining right being servient to the surface to the extent of sufficient support to sustain it in its natural state; and such operator is not liable for any incidental damages necessarily occasioned by the ordinary and careful operation of his mine, and may occupy so much of the surface, adopt such machinery and modes of mining, and establish such auxiliary appliances and instrumentalities as are ordinarily used in such business and as may be reasonably necessary for the profitable and beneficial enjoyment of his property, and he is not limited to such appliances as were in existence when the grant was made to him, but may keep pace with the progress of society and with modern invention.

Bagley *v.* Republic Iron & Steel Co. (Alabama), 69 Southern, 17, p. 19, June, 1915.
See Schobert *v.* Pittsburg Coal & Mining Co., 254 Illinois, 474, 98 Northeastern, 945, 40 L. R. A. (N. S.), 827.
Moore *v.* Indian Camp Coal Co., 75 Ohio State, 493, 80 Northeastern, 6.
Webber *v.* Vogel, 189 Pennsylvania, 156, 42 Atlantic, 4.

USE OF HAULAGEWAYS AND SHAFTS FOR MINING OPERATIONS ON OTHER LANDS—INJUNCTION.

A coal-mine operator who under certain deeds owns the coal thereby conveyed and the right to the possession of the haulageway and the air shaft used in the mining of such coal can not, while continuing the mining, be guilty of a trespass or other wrong by mere occupancy of the haulageway or the shaft or by any use thereof which does not injure or interfere with the use of the nonmineral part of the land in which such coal is found, and can not, therefore, be enjoined in a suit by the surface owner from using the haulageway and air shaft where there is no evidence to show that the injury complained of resulted from the haulage of coal from other lands owned by the operator through the passageway or from the ventilation through the shaft of mines on such other land so owned by the operator.

Bagley *v.* Republic Iron & Steel Co. (Alabama), 69 Southern, 17, p. 18, June, 1915.

EXPERT EVIDENCE AS TO OPERATIONS.

In an action by an injured miner for damages and in controversies over mining property the conditions may be such that experts will be allowed to give expert testimony by way of opinion, because they are presumed to have acquired peculiar skill and knowledge and are more capable of forming a correct opinion as to the subject matter of the question under discussion than inexperienced persons, and their opinions are admitted in evidence for the purpose of aiding the jury in understanding questions which inexperienced persons are not likely to decide correctly without such assistance; but the testimony of such experts may receive only such consideration by the jury as the testimony may appear to the jury to deserve.

American Bauxite Co. *v.* Dunn (Arkansas), 178 Southwestern, 934, p. 936, July, 1915.

RULES REGULATING OPERATIONS—ADMISSIBILITY IN EVIDENCE.

In an action by a miner for injuries received while working in a mine a set of rules promulgated and posted at the mine by the State mining inspector several months after the injury are not admissible in evidence from the mere fact that they were similar to those posted by the defendant prior to the injury, the purpose of which was to show the reasonableness of the rules so posted.

Zelavin *v.* Tonopah Belmont Development Co. (Nevada), 149 Pacific, 188, p. 189, June, 1915.

SURFACE SUPPORT.

MINING AND REMOVAL OF COAL BY OWNER—SUPPORT OF SURFACE.

The owner of an entire estate may grant the surface of the land and reserve the mineral estate, with the right to mine and remove the minerals without liability for injury or damage done to the surface, and in such case the grantor, or those claiming through him, may mine and remove all the coal without being compelled to support the surface.

Kirwin *v.* Delaware, Lackawanna & Western Railroad Co. (Pennsylvania), 94 Atlantic, 468, p. 469, April, 1915.

See Graff Furnace Co. *v.* Scranton Coal Co., 244 Pennsylvania, 592, 91 Atlantic, 508.

DUTY OF OPERATOR TO SUPPORT SURFACE—ARTIFICIAL WEIGHT.

Props sufficient to support the surface of the soil in its original state are all that is required of a mine owner and operator; and where the subsidence of the surface was caused by artificial weight upon the surface of the soil, this is a matter of defense, and it is incumbent upon the defendant to allege such facts if he desires to rely upon them as a defense to the action.

Jackson Hill Coal & Coke Co. *v.* Bales (Indiana), 108 Northeastern, 962, p. 963, May, 1915.

MINING AND REMOVING COAL—LIABILITY FOR INJURY TO SURFACE.

Under a deed conveying the surface of land and reserving the coal and minerals beneath the surface, with the right to mine and remove the same, as well as coal from adjoining lands by any subterranean passage or process for the benefit of the grantor, without incurring in any event whatsoever any liability for any injury or damage which may be caused or done to the surface of such premises, or to any buildings or improvements now erected or which at any time hereafter may be erected thereon, neither the grantee of the surface nor anyone holding under him can recover either from the grantor or from any subsequent grantee of the minerals damages to a house and lot based upon alleged disturbances of the surface caused by mining operations, in the absence of averments and proof that the owner of the mineral was guilty of negligence in the manner of mining and removing the underlying mineral.

Kirwin *v.* Delaware, Lackawanna & Western Railroad Co. (Pennsylvania), 94, Atlantic, 468, p. 469, April, 1915.

Kirwin *v.* Delaware, Lackawanna & Western Railroad Co. (Pennsylvania), 94 Atlantic, 469, p. 470, April, 1915.

FAILURE TO SUPPORT SURFACE—MEASURE OF DAMAGES.

In an action by the owner of the surface against a mine operator for damages caused by the operator's failure to furnish sufficient props to support the surface the measure of damages is the difference between the value of the real estate immediately before and immediately after the subsidence complained of.

Jackson Hill Coal & Coke Co. *v.* Bales (Indiana), 108 Northeastern, 962, p. 964, May, 1915.

LESSEE LIABLE TO SURFACE OWNER.

The person whether owner or lessee who takes the coal out of the mine without leaving proper support for the surface, is the one that is liable for damages to the owner of the surface if the same subsided by reason of his failure to properly support it.

Jackson Hill Coal & Coke Co. *v.* Bales (Indiana), 108 Northeastern, 962, p. 964, May, 1915.

INDEPENDENT CONTRACTOR.

DUTY OF OPERATOR TO EMPLOYEE OF INDEPENDENT CONTRACTOR.

A miner employed by an independent contractor stands to the operator in the relation of an invitee upon the operator's premises to whom, in general, the operator owes only the duty of ordinary care and prudence to keep the premises under his control reasonably safe and under such circumstances the operator assumes to all who accept the invitation the duty to warn them of any danger in coming which he knows of or ought to know of, and of which they are unaware.

Republic Steel & Iron Co. *v.* Luster (Alabama), 68 Southern, 358, April, 1915.
See Sloss-Sheffield Steel & Iron Co. *v.* Hubbard (Alabama), 68 Southern, 571, April, 1915.
Stith Coal Co. *v.* Harris (Alabama), 68 Southern, 797, April, 1915.

RELATION TO OPERATOR A QUESTION OF FACT.

Where a complaint in an action by an injured miner for damages shows that he was engaged in mining coal for the defendant operator and was assisted by others employed by him and that the operator paid him monthly a certain rate for coal mined; that the plaintiff was driving a heading and also keeping up a room, so that when he could not get work in the heading he worked in the room, and where the proof shows that the instrumentalities used in the work, beside the men, were blasting materials, picks, shovels, axes, cars on which the coal was loaded and moved out of the room by the operator onto the main entry tracks, tracks and motors for moving cars; and where it appears that it was the duty of the operator's mine foreman to see that the plaintiff and his assistants were furnished with proper timbers for propping their working places and such mine foreman controlled the miners and looked after the entire mine; and where it was the duty of the superintendent to inspect the roof of the mine at the point where the plaintiff was injured, it becomes under such circumstances, a question of fact for a jury to determine whether the operator had control and supervision of the plaintiff and agencies by which the mining within the area of the plaintiff's labor was accomplished, or whether the plaintiff was rendering service in the course of an independent occupation, and whether the relation was that of master and servant or contractor and contractee.

Stith Coal Co. *v.* Harris (Alabama), 68 Southern, 797, p. 799, May, 1915.

LIABILITY OF OPERATOR FOR INJURY TO CONTRACTOR'S EMPLOYEE.

Where a mine operator employs an independent contractor to mine and remove coal from a completed mine and where such contractor furnishes his own tools, explosives, and employs and discharges at pleasure his own help, and is paid for the coal mined by the town, such operator is not liable for an injury to an employee of such independent contractor, where the injury was caused by a defect in the appliance used by such independent contractor, though the operator furnishing a hoisting engine and cable and a car, for the purpose of hauling the coal out of the mine, but where the manner of operating and adjusting and connecting the engine and cables and car was left to such independent contractor and where the injury was not caused by any defect in the appliances so furnished by the operator.

Republic Steel & Iron Co. *v.* Luster (Alabama), 68 Southern, 358, p. 359. April, 1915.
See Stith Coal Co. *v.* Harris (Alabama), 68 Southern, 797, p. 799, April, 1915.

An experienced coal miner employed by a mine operator under a contract to drive a certain heading from the main entry, and for which he was to be paid by the year for the rock and coal removed, and who hired his own help and did the work in his own way, except that he was responsible to the operator for performing the work according to the rules and regulations of the mine, and who, as a part of the work, was to lay the tracks for the operation of the mine, is an independent contractor, and the operator is not liable to him for an injury received while performing the work under his contract.

Woodward Iron Co. *v.* Wade (Alabama), 68 Southern, 1008, p. 1010, June, 1915.

OPERATOR NOT LIABLE TO CONTRACTOR OPERATING UNDER GENERAL ORDER.

Where an experienced miner was employed by an operator to drive a certain heading from the main center, and for which he was to be paid by the year for rock and coal removed, and where, as a part of his contract, he was to lay the tracks for the operation of the mine, and where such tracks were to be laid on centers, and where it appeared that a part of such tracks was off center, a general order or instruction on the part of the mine operator or its superintendent to such employee to the effect that he must place the tracks on centers as furnished by the engineers and to make the tracks conform to the contractual requirements is a very different thing from ordering a dependent servant to do a particular act of service, and the operator is not liable under such circumstances to the employee for an injury received by him while changing the entry and tracks and making the tracks conform to the contractual requirements.

Woodward Iron Co. v. Wade (Alabama), 68 Southern, 1008, p. 1010, June, 1915.

DUTY OF OPERATOR TO KEEP ROOF OF ENTRIES SAFE—LIABILITY FOR INJURY.

A mine owner and operator is liable for an injury to a miner employed by an independent contractor, caused by a fall of rock from the roof, where the proof shows that the rock which fell upon and injured the miner was from the roof of an entry to the heading in which the contractor and his miners were engaged in mining, and where it appears that the contractor had neglected to prop the roof of the entry in that part of the mine as his miners proceeded with the work of driving the heading, but thereafter it was the operator's duty to look after the condition of the roof and keep it safe; and where it is further shown that the work the operator was engaged in doing had proceeded until this point had been passed a considerable distance and the miner's injuries were caused by the condition of the entry and not the work the independent contractor and his miners were engaged in doing. Under such circumstances it was the operator's duty to maintain the entry where the miner was injured reasonably safe for the benefit of a class of persons to which the miner belonged, and if the miner's injuries resulted from a condition in the roof that was dangerous and could have been made reasonably safe by the exercise of ordinary care, the operator would be liable, whether the injured person was an employee of such independent contractor, as the liability in such case arose out of the breach of duty the operator owed the miner to maintain the roof in a reasonably safe condition and to guard him against unnecessary dangers and not by virtue of any privity of contract between the miner and the operator.

Sloss-Sheffield Steel & Iron Co. *v.* Hubbard (Alabama), 68 Southern, 571, p. 572, May, 1915.

NEGLIGENCE OF OPERATOR OR INDEPENDENT CONTRACTOR—QUESTION OF FACT.

Where it appears that a mine operator furnished the tools, machinery, and other instrumentalities used in the work of mining by an independent contractor, and that the operator's foreman and superintendent reserved the right to discharge such independent contractor or his men at pleasure, and the operator maintained a regular gang of workmen and miners to keep up the entries and maintain the safety of the ways of the mine, and the mine foreman directed the work, the question as to whether an injury to an employee of such independent contractor was proximately caused by negligence and the question as to whether the negligence was that of the mine operator or its servants are questions of fact to be determined by the jury trying the case.

Sloss-Sheffield Steel & Iron Co. *v.* Hubbard (Alabama), 68 Southern, 571, p. 573, May, 1915.

CONTRACTS RELATING TO OPERATIONS.

AGREEMENT FOR ARBITRATION—CONSTRUCTION AND ENFORCEMENT.

An agreement contained in a contract between two mining corporations to the effect that any dispute arising under a contract or charter should be settled at a certain place by arbitration and specifying the method of selecting the arbitrators relates to the law of remedies and not of right, and the law that governs remedies is the law of the forum, and where the forum is a Federal court such court is not bound to follow or conform to the decisions of the State jurisdiction in which it may happen to sit.

United States Asphalt Refining Co. *v.* Trinidad Lake Petroleum Co., 222 Federal, 1006, p. 1011, January, 1915.

AGREEMENT TO WORK A CERTAIN NUMBER OF MINERS—BREACH.

A contract for the transfer of and to work and operate a mine, by which the transferee agrees to work and to have employed in the construction of a tunnel provided for at least four men per day, working 20 days per calender month, is not complied with where less than four men were employed in working in a tunnel, as the agreement to employ at least four men per day does not mean that the transferee can employ two men, each of whom should work 16 hours each day for 20 days in each month, or if they work 30 days of proportionately less hours, as the contract expressly provides for at least four men per day working 20 days in each month and not for 80 shifts to work in each month by less than four men.

Arkoosh *v.* Sorrenson (Utah), 150 Pacific, 959, p. 960, August, 1915.

CONSTRUCTION OF CONTRACT—RIGHT OF OPERATOR TO ORE.

A contract to dig and construct a tunnel provided that the operator was entitled to all the ore he encountered and excavated within the four lines of the tunnel and was given the right to drive other and similar tunnels in any and all portions of the claims described, and to stope any and all veins, lodes, and ledges encountered, does not give the operator the right to ore taken from an old tunnel or stope which had long previously existed on the mining claim and especially where the operator did not claim the ore under a clause in the contract giving him a certain share of the ore mined.

Arkoosh *v.* Sorrenson (Utah), 150 Pacific, 959, p. 961, August, 1915.

NUISANCE—POLLUTION OF WATER.

INJURIES FROM NUISANCE AS CAUSE OF ACTION—PLEADING.

In actions for damages caused by an alleged nuisance maintained by a mine operator, the injurious consequences resulting from the nuisance, rather than acts which produced the nuisance, constitute the cause of action, and the cause of action does not arise until harmful consequences occur, and negligence of the operator is not ordinarily an essential element in such actions. But where the facts are so stated that the conclusion arises as a logical sequence that the harmful consequences complained of proximately resulted from the acts of the owner in the operation of his mines, and the maintenance and use of his washers, is sufficient to show a liability as a matter of pleading.

Yolanda Coal & Coke Co. *v.* Pierce (Alabama), 68 Southern, 563, p. 595, May, 1915.

INJURY TO CATTLE—DUTY TO LESSEN DAMAGES.

In an action against a coal mine operator for damages for polluting a stream and by reason of which the plaintiff's cattle were diseased and many of them died from the effects of drinking polluted water, the plaintiff is entitled to recover such damages as proximately resulted to him in the use of his property for the purpose for which he was using it; and if he was unreasonably deprived of the use of the water, and if his cattle were poisoned by deposits of deleterious matter in the stream by the defendant, and if the death of the cattle proximately resulted therefrom, he would be entitled to recover the reasonable market value of any animals that died from such cause; and if the remaining herd of cattle were, as a proximate result of the defendant's wrong, depreciated in value, he would be entitled to recover whatever such depreciation in value was. But the law imposed upon the plaintiff the duty of taking steps to minimize the damages which were being done to him on account of the nuisance complained of, and if the plaintiff knew that his cattle were being injured by drinking the poisoned water and could have reasonably secured water elsewhere and thereby lessened the damages that were being done, it was his duty to secure such water and lessen such damages, and he can not recover on account of any damages which he could have lessened.

Tenessee Coal, Iron & Railroad Co. *v.* Wright (Alabama), 68 Southern, 339, p. 340, April, 1915.

POLLUTION OF WATER AND INJURY TO LAND—ELEMENTS OF DAMAGES.

Where a mine operator in the operation of its mine places in a stream and the tributaries thereof large quantities of waste, refuse, débris, tailings, culm, and other deleterious or poisonous matters and substances from its mines and other industries, and by reason of which the substances pollute the water of the stream and are washed down into and upon the lands of a lower owner, such landowner may recover damages under an allegation of permanent injury to his land from impairment of water supply, impoverishment of the soil, resulting in depreciation in value, depreciation of rental value, impairment of the right of comfortable enjoyment, injuries to crops, and sickness from noxious odors and poisonous gases.

Yolanda Coal & Coke Co. *v.* Pierce (Alabama), 68 Southern, 563, p. 565, May, 1915.

DAMAGES TO REAL ESTATE—INCONVENIENCES AS PERSONAL INJURIES.

It is sufficient to show a cause of action for damages and to render a mine owner and operator liable, where in an action for damages against such mine owner and operator for the pollution of the waters of a stream, and where the plaintiff's evidence tended to show that from the use of the washers at the mines of the operator and other conditions maintained in the mining operations the water in a stream running through the premises of the plaintiff were polluted; that refuse matter from the mines settled in the bed of the stream; that the water before its pollution was pure and fit for domestic use, and that by the pollution of the stream the water was rendered unfit for use and emitted noxious gases and disagreeable odors that rendered the use of the plaintiff's property less comfortable as a home, depreciated the rental and market value greatly, and put the plaintiff and his family to great inconvenience in obtaining pure water for drinking and laundry purposes and for watering stock, and caused sickness in the family; under the rule that a person who with foul odors and noxious gases renders a man's life unendurable in his home and thus annoys and inconveniences him does him a personal injury as surely as one who commits a battery upon him, and damages thus suffered, like mental pain and anguish, are not the subject of proof by monetary standard but must be left to the sound judgment and discretion of a jury trying the cause, and in assessing damages under such circumstances much latitude and discretion are allowed to the jury, and it is not for the court to say, under such circumstances, that a verdict is excessive.

Yolanda Coal & Coke Co. *v.* Pierce (Alabama), 68 Southern, 563, p. 566, May, 1915.

RIGHT OF ACTION IN THE ABSENCE OF NEGLIGENCE.

It is a matter of common knowledge that pure and wholesome water for domestic purposes and in farming operations is a valuable asset, and its presence or absence materially affects the value of residence and farm properties, and the law is that such rights can not be destroyed by a superior riparian proprietor in the conduct of his mining operations by the pollution of the water in the stream to such extent as to render the water in the stream unfit for domestic use; and to so pollute the stream by such mining operations that waters are impure and unfit for domestic use and from which noxious gases and disagreeable odors arise, rendering the property used as a residence less comfortable, gives a right of action for damages, regardless of the methods of operation, and negligence as applied to the operation is not a necessary predicate to the cause of action.

Yolanda Coal & Coke Co. *v.* Pierce (Alabama), 68 Southern, 563, p. 565, May, 1915.

DEPOSITING SLACK IN STREAM—LIABILITY FOR DAMAGES TO LANDOWNER.

In an action by a landowner against the owner and operator of a coal mine for damages caused by the washing down of slack and fine coal from deposits negligently placed on the bank of a stream by the coal operator, it was not incumbent upon the plaintiff, the landowner, to produce witnesses who could testify that they saw the slack and coal and followed it as it washed down the creek and saw it fill up the channel and cause the injury to the complainant's land as complained of; but it is sufficient to prove that the defendant operated the coal mine on the creek or its tributaries above the complainant's land, and that the defendant, the coal-mine operator, dumped the slack and fine coal into the stream, and that slack and fine coal obstructed the bed of the stream near the complainant's land, thereby causing the water to overflow and damage complainant's land; and the fact, if such fact existed, that other mine operators located between the mine of the defendant and the complainant's land deposited slack or fine coal in the bed of the stream would not relieve the defendant from liability, as the defendant can not escape liability merely because the complainant does not or can not apportion the damages between the wrongdoers.

North Jellico Coal Co. *v.* Trosper (Kentucky), 177 Southwestern, 241, p. 243, June, 1915.

SUCCESSIVE ACTIONS FOR LOSS OF RENTS—RENTS MERGED IN DAMAGES FOR TOTAL INJURIES.

Where as the result of the permanent injury to real estate caused by the operation of coal mines and the dumping of slate and shale from the mine and discharging thereon quantities of contaminated and poisonous water, resulting in the pollution of the waters of a stream, spring, and well on the premises and impoverishing the soil, rendering it less fertile and productive, the rental value of such property has depreciated or has been destroyed, and where the owner has not recovered damages for the permanent injury caused by the nuisance, the loss of rents is a constantly recurring injury and may be recovered in repeated actions until the nuisance is abated; but if the nuisance has resulted in the total destruction of the estate, rendering it worthless, and damages for the permanent injury have been recovered and the owner made whole, his loss by way of rents is merged in the recovery for the permanent injury, and he can not recover in any subsequent action.

Pratt Consolidated Coal Co. *v.* Morton (Alabama), 68 Southern, 1015, p. 1016, April, 1915.

DAMAGES FOR PERMANENT OF TEMPORARY INJURIES—PROOF OF VALUE AND DAMAGES.

Where real estate is permanently injured by a nuisance resulting from the operation of coal mines, damages for the permanent injury are recoverable in the same action with injuries of a temporary nature such as rents or profits, and where the nuisance is a continuing one and damages for permanent injuries may be barred by the statute of limitations, proof of the value of the property before any injury resulted to it from the nuisance is admissible, and its value after the injury as a predicate for the recovery of the constantly recurring damages resulting from the nuisance; but it is not permissible to prove, as showing the market value of the land, the price paid several years prior to the alleged injury, but the criterion in establishing permanent injury is the difference in the market value before the injury and after the lands had suffered the injury.

Pratt Consolidated Coal Co. *v.* Morton (Alabama), 68 Southern, 1015, p. 1016, April, 1915.

PREVENTING OPERATIONS—INJUNCTIONS.

INJUNCTION AGAINST OPERATING—DISSOLUTION ON FILING BOND.

An injunction obtained by the owner of land against a railroad company by which the company is enjoined from boring oil wells on its right of way, on allegations that the railroad company has only a right of way and not a fee in the land, and that such boring is a trespass and a disturbance of the oil under adjoining lands of complainant, and that the complainant will be injured by the continued boring, will be dissolved on bond properly executed by the defendant railroad company, in the discretion of the trial court, where the bond is conditioned for all damages sustained by the plaintiff in case of a definitive judgment against the defendant in the final trial of the suit.

Natalie Oil Co. *v.* Louisiana Railway & Navigation Co. (Louisiana), 69 Southern, 146, June, 1915.
See Hayne *v.* Edenborn (Louisiana), 68 Southern, 787, May, 1915.

NO RIGHT TO JURY TRIAL.

An action by a mine owner and operator to enjoin an adjoining owner from mining and removing ore of a vein apexing in the plaintiff's mining claim is equitable in character, and the damages sought are merely incidental. The cause is properly triable to the court, and neither party has a right to demand or have a jury; and where the issue is a question of apex right, involving a question of fact and no question of law, except in so far as the determination of what constitutes a vein might be so considered, and where the court made its finding and entered judgment after hearing the evidence and on an inspection of the premises, a court on appeal will not disturb the verdict and judgment.

Esselstyn *v.* United States Gold Corporation (Colorado), 149 Pacific, 93, page 94, June, 1915.

DUMPING REFUSE ON SURFACE—INJUNCTION—REMEDY AT LAW.

Where a coal-mine operator has been dumping refuse from other mines upon the lands of the complainant, and where the operator ceased such work on the request of the landowner and there was no intention or danger shown of a repetition of the injury, there can be no injunction granted, as the remedy in such case is complete and adequate at law.

Bagley *v.* Republic Iron & Steel Co. (Alabama), 69 Southern, 17, June, 1915.

NEGATIVE COVENANTS—IMPROPER USE OF SURFACE BY LESSEE—INJUNCTION.

A lease of Indian lands for mining coal and asphalt in compliance with an act of Congress relating to mining leases in the Creek Nation in addition to the ordinary provisions for mining and the usual stipulations and covenants, contained a provision to the effect that the demise and lease was for the sole purpose of prospecting for and mining coal and asphalt, and the lessee was to occupy so much only of the surface of land as might be reasonably necessary to carry on the work of prospecting for, mining, storing, and removing the coal and asphalt mined, with an agreement on the part of the lessee to commit no waste, and to suffer no waste to be committed, upon the premises, and that the lessee would not use the premises, nor suffer them to be used, for any other purpose than that authorized in the lease. The lease, by its provisions, did not become valid until approved by the Secretary of the Interior, and was at all times subject to his rules and regulations relative to mineral leases in the Creek Nation. The lease contained no provision, either express or implied, giving the lessee the right to use the surface of the demised premises as a dumping ground for shale and other waste taken from mines upon premises other than those described in the lease, nor is it reasonable that the Secretary of the Interior would approve a lease which, properly construed, would permit a lessee to use the premises for such purposes, and where it appeared that the lessee had sunk shafts upon the premises and had extended tunnels from these into adjacent lands from which it was mining large quantities of coal on the leased premises and was dumping large quantities of shale and waste brought from such adjacent lands upon the surface of the leased premises and was occupying a large part of such surface with buildings and dwellings for the use of employees engaged in such outside mining operations. The use of the premises leased was restricted by the express terms of the lease to prospecting for and mining coal and asphalt on the premises, and in such case the law implies a negative covenant not to use the premises for any other purposes than those specified; and a court of equity will, by injunction, protect the lessor against the use by the lessee of the premises in violation of such negative covenant.

Sharum *v.* Whitehead Coal Mining Co., 223 Federal, 282, page 289, April, 1915. See Consolidated Coal Co. *v.* Mary Ann Schmisseur, 135 Illinois, 371, 25 N. E., 705.

The execution of a lease to mine coal and asphalt under the lands described will not authorize the lessee to dump slate and refuse upon the surface of the demised lands unless the right is expressly granted; and where such a lease does not authorize the lessee to use the openings, shafts, and tunnels on the demised lands to mine adjoining property not belonging to the lessor, the lessee takes no right except under the lease, and the failure of the lease to grant such privilege impliedly negatives the privilege, thus raising an implied negative covenant which is analogous to specific performance that will be enforced by injunction.

Sharum *v.* Whitehead Coal Mining Co., 223 Federal, 282, page 290, April, 1915. See Brasfield *v.* Burnwell Coal Co., 180 Alabama, 185, 60 Southern, 382.

MINING LEASES.

LEASES GENERALLY—CONSTRUCTION.

FALSE REPRESENTATIONS TO AGENT—RIGHT TO RESCIND.

An action to rescind a contract of purchase of a mining lease on the ground of fraud and false representations can not be maintained and the contract can not be rescinded where it is shown that the negotiation for the contract and an examination in the purchaser's behalf was made by an expert miner of long and varied experience, and where it clearly appears that the representations in part did not deceive the agent and where other representations were not relied upon by the agent, and where it clearly appears that the agent acted on his own judgment and did not accept "seriously" the statements and representations made to him by the seller, as the rule is that an attempt to fraudulently deceive must be so far successful as to have induced belief and reliance on the part of the person claiming to be injured.

Greenstreet *v.* Walsch (Missouri Appeals), 176 Southwestern, 1062, page 1063, May, 1915.

DIAMOND LEASE—ACTION TO RECOVER DIAMONDS ON GROUND OF FRAUD.

The lessor of lands leased for the purpose of mining for diamonds can not in an action of replevin recover possession of diamonds mined by the lessee and belonging to him under the lease, on the ground that the lease was procured for the fraudulent purpose of discrediting the mine and keeping the lessor in the dark as to the value of diamonds discovered and to stifle the business of mining, depress the value of the land and buy it for a minimum price, in the absence of proof of the alleged fraudulent acts by the preponderance of the testimony; and the mere failure of the lessee to perform some stipulations and conditions of the lease, in the absence of proof of fraud, would not entitle the lessor to recover in such an action.

Mauney *v.* Millar (Arkansas), 175 Southwestern, 402, March, 1915.

COAL LEASES.

PRACTICAL CONSTRUCTION—MINIMUM AMOUNT OF DIFFERENT GRADES.

A coal lease provided that the lessee should pay for all coal mined above the size of pea coal at the rate and price of 25 cents per ton of 2,240 pounds, and for pea coal 12½ cents per ton royalty until it is worth within 25 cents per ton of chestnut coal, and then to be 25 cents per ton, payments of royalty to be quarterly for the first two years and for not less than 4,500 tons each year; for the third year, 9,000 tons; for the fourth year, 13,500 tons; and for each year thereafter, 18,000 tons; and it was agreed that if the lessee in any one year paid for more coal than mined he had the right at any time thereafter to mine the coal paid for without further charge. There was no provision specifying what proportion of the several sizes should compose the minimum quantity to be mined annually by the lessee, and the contract in this respect was left ambiguous, and the lease must be construed in this doubtful aspect as to what proportion of the several sizes shall compose the minimum quantity to be mined annually; and the parties in this respect have the right to invoke the aid of contemporaneous construction; and, applying this rule, the minimum product was to be determined by the quantity of coal of ordinary size produced at the mine, and for more than 30 years the lessee paid to the lessor and the lessor received and receipted for the royalty on the minimum product on this basis, the parties thereby placing their own interpretation upon the lease; and they have thus determined by their long course of conduct what was in their minds when they wrote and signed the lease; and a court is not at liberty after a period of more than 30 years to declare that the parties had a different intention from that shown by their own interpretation of the lease; and a court must conclude that the parties intended that the minimum amount of royalty should be computed on coal of prepared size and not on such size and pea coal.

Lehigh Valley Railroad Co. *v.* Searle (Pennsylvania), 94 Atlantic, 74, p. 75, March, 1915.

DISAGREEMENT AS TO ROYALTIES DUE—FORFEITURE NOT PERMITTED.

A coal lease providing that on failure to pay any installment of royalty for 90 days, the lease and all rights under it shall, at the option of the lessor, become forfeited, can not be, at the option of the lessor, forfeited for a small amount of royalty due under the lessor's construction of the lease; and where the failure to pay arose from the difference in the interpretation put upon the lease by the parties, and where the interpretation by the lessor was contrary to the practical construction placed upon the lease by the parties for a period of over 30 years, as the forfeiture clause manifestly never was intended to enforce a forfeiture under such circumstances, and it would be against reason and good conscience to permit the annulment of a lease where the forfeiture clause was intended to enforce payment of royalty due by the terms of the lease, and until that was legally ascertained, if reasonably in doubt, there could be no just nor conscionable ground on which it could be permitted to operate. Equity will not permit a party to a contract to enforce it or to compel the forfeiture by means manifestly never intended and such as are clearly harsh and oppressive, and the rule in equity is that where a penalty or forfeiture clause is designed merely as a security to enforce the principal obligation, it is as much against conscience to permit a party to pervert it to a different and oppressive purpose as it would be to allow him to substitute another for the principal obligation.

Lehigh Valley Railroad Co. *v.* Searle (Pennsylvania), 94 Atlantic, 74, p. 77, March, 1915.

OIL AND GAS LEASES.

NATURE OF LEASE—RECORDING NECESSARY.

An oil and gas lease is a chattel real as contradistinguished from real property, and while it merely grants the right to prospect for oil and gas, vesting no title in the land itself and no title to the oil and gas until reduced to actual possession, it is required by the statute to be in writing, acknowledged and recorded in order to be valid against third persons.

Tupeker *v.* Deaner (Oklahoma), 148 Pacific, 853, May, 1915.

CONSTRUCTION—AREA FOR OPERATING WELL—PAROL PROOF INADMISSIBLE.

In an action involving the construction and the rights of the parties under an oil and gas lease and for a forfeiture of the same which gave to the lessee the necessary space to operate a well, in a case of forfeiture the lessee is not entitled to prove a parol agreement to the effect that he should be entitled to an acre of land for each well opened.

Moore *v.* Decker (Texas Civil Appeals), 176 Southwestern, 816, p. 817, May, 1915.

CONSTRUCTION AND CONSIDERATION—IMPLIED COVENANT TO OPERATE—FORFEITURE.

A lease or conveyance of all the oil and gas and other minerals in and under certain described lands for a stated consideration, giving the right to enter and drill and operate for oil, and providing for a stipulated royalty or portion of the oil produced, and providing that in case operations are not commenced and prosecuted with due diligence on or before a certain stated date, then the grant should immediately become null and void as to both parties, and if oil and gas or other minerals shall be discovered the grant or conveyance shall be in full force and effect for 25 years from the time of discovery and as much longer as oil, gas, or other minerals can be produced in paying quantities, is satisfied by commencement of operations within the stated time, and the discovery of oil by an assignee of the grantee or lessee inured to the benefit of both assignee and lessee and to the benefit of the tract of land subleased as well as to the remaining original leased tract, and to that extent had the effect of vesting the right of the original grantee or lessee in the assignee or sublessee, and the assignee of the lessee thereby became the tenant of the lessor by agreement for the specified term of 25 years and not at will and could not be divested of this vested right at the will of the lessor. But the conveyance or lease must be held to contain an implied obligation, if not expressed, that the lessees would continue to mine and operate the lease for the production of oil and gas during the time of its continuance, and that as a part of the consideration for the lease, one-eighth of the oil produced should be delivered to the lessor; and this and other provisions clearly evidence the purpose not only to prospect for and discover oil but to produce, transport, and drill for oil, and pipes were to be laid for such purpose, and the mere discovery of oil did not require the performance of all the provisions set out. The agreement implies that the lessee or his assigns will produce oil upon the discovery during the term and will pay the stipulated royalty, and a failure on the part of the lessee or his assigns to perform this implied obligation or condition subsequent would work a forfeiture of the lease. But a mere temporary cessation of work in operating or drilling on the land for oil should not work a forfeiture of the lease in consideration of the very large sum paid in the first instance and large expenditures made by the assignees in drilling wells; and the evidence to justify a forfeiture should show more than the abandonment of the wells when they ceased to produce and it would be unreasonable to require a continued pumping of a well which had ceased to pay anything or to leave the casing in a well in which there had never been any oil. But if the lessee or his assigns discovered oil and prosecuted their discovery with diligence and in good faith, though they should exhaust their well or abandon one producing no oil, a forfeiture should not be declared as a matter of law, and the mere cessation alone ought not to produce a forfeiture unless the proof clearly showed an abandonment of the lease.

Fisher *v.* Crescent Oil Co. (Texas Civil Appeals), 178 Southwestern, 905, June, 1915.

CONSTRUCTION—RIGHTS AND LIABILITIES OF LESSOR AND LESSEE.

An oil-and-gas lease containing a provision of forfeiture on failure of the lessee to perform its conditions is for the benefit of the lessor only, and he can either avail himself of such failure under the lease or he can waive the forfeiture and collect the rentals stipulated in the lease unless there is an express stipulation to the effect that the lease shall become null and void unless the lessee shall either drill within the stated time or pay the rental, or words of similar import.

Cohn *v.* Clark (Oklahoma), 150 Pacific, 467, p. 471, June, 1915.

LESSEE PROTECTED BY JUDGMENT.

Where an oil-and-gas lease gave to the lessee the use of such space as is necessary to operate a well the lessee can not complain of a judgment giving the possession of the land upon which the well was drilled to the lessor, the judgment expressly recognizing and giving to the lessee the use of such space as is necessary to operate the well drilled by the lessee.

Moore *v.* Decker (Texas Civil Appeals), 176 Southwestern, 816, p. 817, May, 1915.

LESSOR ENJOINED FROM EXECUTING SECOND LEASE.

Where the holder of a valid oil-and-gas lease has obtained vested rights by drilling wells and by the production of oil and gas, equity will enjoin the lessor from creating a cloud on his title by executing to a stranger another lease on the same property where it appears to be reasonably certain that such cloud will be created unless enjoined.

Castlebrook Carbon Black Co. *v.* Ferrell (West Virginia), 85 Southeastern, 544, p. 546, May, 1915.

ASSIGNEE ACCEPTING ASSIGNMENT WITH NOTICE OF PRIOR LEASE.

In the assignment of an oil-and-gas lease of all the right, title, and interest of the lessee, an assignor therein, there is no implied warranty of title, and an assignee who accepts such assignment with knowledge of an existing prior lease on the same premises can not complain and can not recover the money paid for the assignment or damages because of the existence of such prior lease.

Tupeker *v.* Deaner (Oklahoma), 148 Pacific, 853, May, 1915.

ASSIGNMENT—EFFECT AND LIABILITY OF ASSIGNEE.

Under an assignment of an oil-and-gas lease, stating that it is intended to convey and does convey to the assignee an undivided one-eighth interest in the working interest of the lease without any obligation on the part of the assignee to the assignor, by virtue of a certain prior contract and assignment, and reciting further that the assignment is made subject to the terms and conditions of the lease, the words "subject to" are words of classification and not of contract, and the assignee did not enter into a personal obligation to fulfill any of the terms of the lease but simply took a one-eighth interest charged with the burden placed upon him by the terms of the lease and was not liable personally for any more than one-eighth of the expenses incurred in development of the oil-and-gas lease.

Cox *v.* Butts (Oklahoma), 149 Pacific, 1090, p. 1091, June, 1915.

LIABILITY OF ASSIGNEE FOR RENT.

Where a lease of land for oil and gas provides that a certain sum shall be paid each year as royalty on the gas produced from each well and marketed off the premises, and where it appears that the lessee operates the lease, markets the gas from wells thereon for a portion of the year, and thereafter assigns the lease, the assignee, in the absence of a special contract, is not liable for the royalties accruing on the wells, the product of which was marketed prior to such assignment of the lease, regardless of the particular time when such royalties became due and payable under the terms of the lease, but in such case the original lessee and assignee is liable for the royalties that accrued during the time he so marketed the product and enjoyed the estate.

Columbus Gas & Fuel Co. *v.* Knox County Oil & Gas Co. (Ohio), 109 Northeastern, 529, p. 530.

LIABILITY OF ASSIGNEE FOR RENTS—RELATION AND LIABILITY OF LESSEE.

The lessee of an oil and gas lease may be liable by the terms of the lease for the rents and royalties accruing during the entire term of the lease; and if the lease is assigned, in the absence of a contract assuming further liability, the assignee is liable only for the rent accruing after the assignment; and as to such rent or royalty accruing after assignment the original lessee stands in relation of surety to the assignee, and under the statute of Ohio may maintain an action against such assignee for any rent or royalty accrued and due and unpaid after the assignment of the lease.

Columbus Gas & Fuel Co. *v.* Knox County Oil & Gas Co. (Ohio), 109 Northeastern, 529, p. 530.

ACTION BY ASSIGNEE TO QUIET TITLE—ASSIGNOR NOT NECESSARY PARTY.

The original lessee and assignor of an oil and gas lease is not a necessary party defendant to an action by the assignee of an ordinary oil and gas lease, brought to quiet his title and to enjoin the lessor from executing a second lease on the same land, where the assignment is absolute and unconditional, under the rule that where an assignment is absolute and unconditional leaving no equitable interest whatever in the assignor, and the extent and validity of the assignment is neither doubted nor denied and there is no remaining liability in the assignor to be affected by the decree, he is not a necessary party.

Castlebrook Carbon Black Co. *v.* Ferrell (West Virginia), 85 Southeastern, 544, p. 545, May, 1915.

COVENANT TO DRILL WELLS OR PAY FORFEIT—SINGLE PENALTY.

A covenant in an oil and gas lease absolutely binding the lessee to complete a well on the premises within four months and providing conditionally that he should complete three more within successive periods of three months, unavoidable delays after starting to drill excepted, and providing that upon failure to drill and complete the wells or any of them the lessee shall pay $100 forfeit for each well specified which he had not then completed, or surrender the lease for cancellation, imposes one penalty for the nondrilling of each well and not successive penalties for each failure to be paid every three months.

Petty v. United States Fuel Gas Co. (West Virginia), 85 Southeastern, 523, May, 1915.

FAILURE TO OPERATE—RECOVERY OF RENTAL.

Under an oil lease providing that the lessee should commence operations in drilling for oil at a particular date, or thereafter pay to the lessor a stipulated sum per acre per annum until a well is drilled, or the lease shall be null and void, the lessor may, upon the lessee's failure to begin operations by drilling for oil within the stipulated time, maintain an action for the stipulated rental until a well is drilled or until the lease is terminated or canceled under the surrender clause contained therein.

Cohn *v.* Clark (Oklahoma), 150 Pacific, 467, p. 469, June, 1915.

DAMAGES FOR FAILURE TO DELIVER LEASE—INSUFFICIENT FINDINGS.

In an action for damages by a complainant against an oil company as the owner of lands for the failure of such oil company to deliver to the complainant a lease supported by a fee title to certain oil lands, together with an oil well assumed to be located thereon, wherein the complainant alleged that the defendant landowner represented that it was the owner in fee simple title of the real estate described and that there was located thereon a certain oil well capable of producing 50 barrels per day and representing that there was located adjacent to such land, and which would be available for the complainant's use, a standard drilling rig, boiler, and engine in good condition, with a complete outfit of drilling tools and a large footage of casing; and alleging further that he had paid on account of the rental reserved a large sum of money, and that the drilling rig, boiler, engine, and drilling tools were not in good condition and that the oil company refused to deliver a lease to 160 acres of proven oil land of which it was vested with a fee simple title, a judgment for $5,200 damages can not be sustained where the findings of the court are not definite and do not show the separate items of damages making the sum total, although the findings are definite as to some of the particular items of damages; and the judgment for the return of the money paid as part consideration of the lease can not be upheld where the action did not proceed upon the theory of rescission, and where there was no offer to rescind or return the consideration and where it is not alleged that the lease which the complainant obtained was without value.

Hullinger *v.* Big Sespe Oil Co. (California), 151 Pacific, 369, August, 1915.

NO TIME FIXED FOR PAYMENTS—REASONABLE TIME IMPLIED.

An oil and gas lease providing that in case no well is commenced within 90 days the lease should be null and void, unless the lessee should pay for the delay at the rate of 50 cents a day until a well was commenced, can not be canceled by the lessor where he had accepted payment after the default of 101 days and the payment of 50 cents a day fixes the rate and not the times of payment, and under such an agreement payments at reasonable times for a default in the failure to drill is sufficient to preserve the lease; and payments quarterly are to be regarded as reasonable.

Smith *v.* Steele (Kansas), 150 Pacific, 519, p. 520, July, 1915.

APPLICATION OF FORFEITURE CLAUSE—RENTALS DISTINCT FROM ROYALTIES.

A clause in an oil and gas lease to the effect that the failure of the lessee to complete a well upon the premises described within the time specified or to pay the rentals at the time and in the manner as therein provided shall ipso facto work a forfeiture of the lease without notice applies only to rentals provided to be paid for delay in drilling and not to rentals or royalties to be paid for gas from a producing well.

Castlebrook Carbon Black Co. *v.* Ferrell (West Virginia), 85 Southeastern, 544, p. 545, May, 1915.

ABANDONMENT AND FORFEITURE.

The distinction between "forfeiture" and "abandonment" as applied to oil and gas conveyances and leases is so shadowy that in discussing the one necessarily the conditions of the other are involved; but one distinction is that "abandonment" rests on the intention of the parties, while forfeiture does not rest upon intent to release the premises, but is an enforced release, and abandonment in this sense is the relinquishment of a right, and a vested title can not ordinarly be lost by abandonment unless there is satisfactory proof of an intention to abandon, and the existence of an intent to waive or abandon the right to drill for oil and gas under the lease is a question of fact, and the lessor must show an intention on the part of the lessee to abandon the lease; and if the proof would authorize the conclusion that there was no such intention, then a court would not be justified in decreeing a forfeiture of the lease. The fact that a lessee had abandoned work on a dry well and removed therefrom the casing is not sufficient evidence of the abandonment of a vested right under a voidable lease, nor is the intention to abandon established by the further fact that the lessee was afterwards unfortunate enough to drill a dry hole. Under a lease for a specified term and a stated valuable consideration paid the lessee may continue his efforts to discover oil and gas or mineral by continued diligent drilling during the entire term of the lease.

Fisher *v.* Crescent Oil Co. (Texas Civil Appeals), 178 Southwestern, 905, p. 908, June, 1915.

RIGHT OF LESSEE TO DRILL ON RAILROAD RIGHT OF WAY—APPEAL FROM INJUNCTION.

An oil and gas lessee authorized to drill and operate for oil and gas on the right of way of a railroad company is entitled to appeal from a judgment enjoining operations under the allegation that the lessee is the lessee of the railroad company which acquired its rights and its right of way only by virtue of the power of eminent domain and for railroad purposes, and that the drilling of an oil well for personal gain is not for such a purpose and will inflict injury upon the complainant by draining oil from his adjoining lands.

Hayne *v.* Edenborn (Louisiana), 68 Southern, 737, p. 738, May, 1915.

LEASE EXECUTED PENDING SUIT—RIGHTS OF LESSEE.

An oil and gas lessee who acquires his lease from a party to a pending suit can not acquire any interest in the land during the pendency of such suit from a party to the suit that would defeat any decree the court might make, but such lessee is bound by the court's decree as if a party to the cause, and the fact that he may have acquired the lease without actual notice of the claim of the plaintiff in the pending suit is not effective as a matter of law, and such lessee is not entitled on his application to be made a party to the suit for the alleged purpose of protecting his interest.

Taylor *v.* Taylor (West Virginia), 85 Southeastern, 652, p. 654, June, 1915.

MINING PROPERTIES.

TAXATION.

MINING CLAIMS SUBJECT TO TAXATION.

The land upon which an unpatented lode-mining claim is located may not be taxed, for the title to the land is in the United States; but the right of possession of such a mining claim is property of great value and is distinct from the land itself, and is therefore subject to taxation. In such case the land on which such mine is located is not assessed for taxes, but the claim itself, the right of possession of the land for mining purposes is the property that is assessed, and a State has the power to tax such interest in a mining claim and to enforce the collection of the tax by a sale.

Earhart *v.* Powers (Arizona), 148 Pacific, 286, p. 287, May, 1915.

MINING CLAIMS AS PERSONAL PROPERTY.

For the purpose of taxation the term "personal property" includes any interest or equity in a valid claim to nonpatented mining claims, either lode or placer.

Earhart *v.* Powers (Arizona), 148 Pacific, 286, p. 288, May, 1915.

RIGHT OF POSSESSION AS DISTINCT FROM LAND.

The inclusion of a "mining claim" in the term "personal property" as provided by section 4847 of the statutes of Arizona (Civil Code of Arizona, 1913) is clearly a recognition by the legislature that the right of possession, as distinct from the land itself, is made the subject of taxation and must be assessed at its full cash value, and there is no provision either in the constitution of Arizona or in the legislative enactments that makes this species of property called "mining claims," which is capable of private ownership, not taxable.

Earhart *v.* Powers (Arizona), 148 Pacific, 286, p. 288, May, 1915.

MINES AND MINING CLAIMS—POSSESSORY RIGHT.

By the term "mining claim" the possessory title is indicated, and by the term "mine" the title to the land as distinguished from the mere possessory right thereto is shown; and the use of the words "mines and mining claims" in the statute governing the subject of taxation distinguishes between the cases in which there is ownership of land, and therefore perfect title to the land, and those cases wherein the miner has the exclusive possession and enjoyment of the land for mining purposes.

Earhart *v.* Powers (Arizona), 148 Pacific, 286, p. 287, May, 1915.

FRANCHISE TO PROSPECT METAL LAND TAXABLE.

A conveyance by which the grantor conveyed to the grantee all the oil, gas, coal, and other minerals in and under a particular tract of land described for a valuable consideration in a stated amount and certain stipulated royalties, with the exclusive right of ingress and egress for the purpose of drilling, mining, and operating for such minerals and the conduct of all operations, and the erection of appliances and structures, the laying of all pipe lines necessary for the production, mining, storing, and transportation thereof, and providing further that the grantee should begin operations within one year from the time of the delivery of the instrument, creates an estate that is subject to taxation against the owner of the fee as a part of the land, the same as any other valuable right or privilege under the statute of Texas (Art. 7504), making taxable all the rights and privileges belonging to or in any wise appertaining to real property, and of mines, minerals, quarries, and fossils in and under the same.

Texas Co. *v.* Daugherty (Texas), 176 Southwestern, 717, May, 1915.

EXEMPTIONS TO STIMULATE MINING.

Article 10, section 1, of the constitution of Nevada, as originally adopted, exempted all mines from taxation; but this section was amended so as to permit assessments for taxation of all patented mining claims, but any patented mining claim upon which $100 worth of work is annually done is made exempt from taxation, and the purpose of this change was to stimulate mining.

Goldfield Consolidated Milling, etc., Co. *v.* Old Sandstrom Annex Gold Mining Co. (Nevada), 150 Pacific, 313, p. 316, July, 1915.

CONSTITUTIONAL PROVISIONS—CLASSIFICATION.

Article 10, section 3, of the constitution of Colorado, adopted in 1876, provided that mines bearing precious metals should be exempt from taxation for 10 years from the adoption of the constitution, and subsequently subject to taxation. Subsequently the legislature, after obtaining the opinion of the supreme court, in 1887, passed an act providing for the taxation of mines and for this purpose divided them into two classes, producing and nonproducing, and all mines producing mineral exceeding in value $1,000 per annum were arbitrarily classed as producing mines. The statute also provided that all producing mines should be valued for revenue purposes at a sum not to exceed one-fifth of the gross proceeds from any mine during the preceding year; and this act was declared constitutional and remained the law until 1902.

Tallon *v.* Vindicator Consolidated Gold Mining Co. (Colorado), 149 Pacific, 108, p. 113, June, 1915.

CONSTRUCTION AND VALIDITY OF STATUTE—ARBITRARY RULE.

The legislature of Colorado, in 1902, amended the act of 1887 providing for the taxation of mines and provided that all mines whose gross production exceeds $5,000 per annum should be classed as producing mines and all others as nonproducing, and that all producing mines should be valued for taxation at a sum equal to one-fourth the gross proceeds for the preceding year, unless the net proceeds exceeded one-fourth of such gross proceeds, and in that event the mine should be valued at an amount equal to the net proceeds for the fiscal year. This statute changed the act of 1887 in three particulars:

1. Producing mines were raised from $1,000 to $5,000. 2. The fraction was raised from one-fifth to one-fourth. 3. Where the net exceeded one-fourth the gross proceeds, then the mine was to be assessed at a sum equal to the net instead of a fraction of the gross proceeds; and this arbitrary rule as to both the acts of 1887 and 1902 has been held constitutional and valid.

Tallon *v.* Vindicator Consolidated Gold Mining Co. (Colorado), 149 Pacific, 108, p. 113, June, 1915.

CONSTITUTIONALITY OF ACT—METHOD OF FIXING VALUE FOR TAXATION.

The legislature of Colorado, in 1913 (Laws of 1913, p. 566, sec. 2), amended the act of 1902, providing for the taxation of mines and required the local assessor to value each producing mine at a sum equal to one-half of the gross proceeds, plus all the net proceeds for the preceding year; and, if it was constitutional in 1887 for the legislature to choose one-fifth of the gross and in 1902 one-fourth of the gross, unless the net exceeded one-fourth of the gross, in which event all the net should be used, it was constitutional in 1913 for the legislature to adopt one-half the gross and all the net proceeds as the criterion or basis for the assessment of producing mines. The legislature did not intend that the fractions mentioned in these different statutes should arbitrarily represent the net proceeds, as in the act of 1902 it provided that the net proceeds should be taxed only if it exceeded one-fourth the gross proceeds, and the act provides the gross proceeds shall be obtained by deducting the cost of transportation and treatment, and the net proceeds shall be ascertained not by an arbitrary fraction of the gross but by deducting from the gross the cost of reduction, and then that a fraction of the gross plus all the net obtained in this way shall be a sum equal to the value of the mine for taxation; and, while the legislature could not say that one-half the gross proceeds plus all the net in fact equals the net proceeds, yet it could lawfully say that the amount so determined should represent the value of the mine for taxation, and in this way it provides a rule for arriving at the value of producing mines for taxation and is constitutional.

Tallon *v.* Vindicator Consolidated Gold Mining Co. (Colorado), 149 Pacific, 108, p. 114, June, 1915.

CONSTITUTIONALITY OF STATUTE—DISCRIMINATION IN TAX SALES OF MINERAL LANDS.

The statute of Wisconsin, section 1042j, Laws of 1913, provides for the taxation of all rights and reservations to enter upon and take away any mineral from any described lands where the title to such right of reservation is vested in any person or corporation other than the owner of the fee and requires the same to be separately assessed for taxation; and provides also that any such reservations and rights may be sold for nonpayment of taxes, but limits the purchasers at such tax sale to the owner of the fee to which the right or reservation is attached or to the State; and provides also that the right or reservation acquired by the State shall not be completely alienated or sold, but may be leased for limited periods of time on a royalty basis. This statute must be treated as a taxing statute enacted for the purpose of raising revenue for governmental purposes and as including within its scope all cases in which by exception, reservation, or express grant the title to ores and mineral land, together with the right of exploring for and mining the same, are vested in some person or persons other than the person or persons who own title to the land and its beneficial use for any and all purposes. The existence of valuable ores or minerals beneath the surface undisclosed is not easily ascertainable but may be known to the owner by drilling and testing the land; and this is a distinction cognate to the purpose of the statute which would support the discrimination found in the statute requiring such owner to furnish the assessor an affidavit of value, while the owners of other estates in the same tract of land are not required to do so. If this were the only objection to the statute, it would be held valid either under the Federal or State Constitution. But the fatal defect in the statute is in the method of acquiring title at tax sales. By other statutes sales of real and personal property of a delinquent taxpayer are subject to competitive bidding at public auction, but under the statute in review the property mentioned is returned delinquent, advertised for public sale, bidders are invited, and the property in all respects, except with reference to the mode of assessment, as other real property; but the invited bidders are prohibited from bidding because they can not become purchasers, and no one but the State or county or the person who owns the remaining interest in the land can bid at the sale. Accordingly, if this is a taxing statute, the purpose of which is to raise revenue for the State expenses, this limitation of bidders is not germane to but in contravention of the purpose of the statute, and discriminates unjustly against the holder of the particular property described, because he loses the advantage of having some one purchase his mineral rights without limitation or restriction. This discrimination is wholly arbitrary and is without legitimate classification on which it can rest, as it does not aid the State in collecting its revenue to limit the number of bidders, nor to require the whole land to be sold, instead of the smallest area, as required by other statutes. While the rule of taxation does not extend to all steps in enforcing collection of the tax, yet it does extend to those essential steps which are necessary parts of the tax proceedings and collection by demand or collection by enforcement process are necessary steps in enforcing the collection of the tax.

State *v.* Donald (Wisconsin), 153 Northwestern, 238, p. 239, June, 1915.

TAXATION OF MINES AND NET PROCEEDS—CONSTITUTIONALITY.

The Colorado statute of 1913 is not unconstitutional on the ground that it results in inequality and injustice between the owners of producing mines as a class because of the method of arriving at the proceeds; but it is neither the net nor the gross proceeds that are taxed, and these are only employed in formulating a rule by which the value of the mine, the only thing taxed, may be established or approximated for the purposes of taxation, and the fact that there may be low-grade and high-grade mines and the difference in the cost of production and extraction are too remote to annul the statute. The fact that mining operations are attended with so many intricate difficulties, uncertainties, and complications, any mode provided for taxation may produce many inequalities and hardships in particular cases, and the ingenuity of man can not devise a system for the taxation of mines in which this will not be the case. The very character of the property is such that there can be no system evolved that in some instances will not produce inequality and lack of uniformity in taxation, and these inequalities must be treated as an unavoidable incident to the ownership of such property. It follows that the act of 1913 is constitutional and provides the rule for establishing the value of producing mines for taxation in the State of Colorado.

Tallon *v.* Vindicator Consolidated Gold Mining Co. (Colorado), 149 Pacific, 108, p. 115, June, 1915.

GROSS PROCEEDS.

The words "gross proceeds" as applied to the taxation of mines and as a basis of taxation mean the amount of money actually received by the mine owner for his ore after deducting the cost of transportation to the place of sale or reduction, and the cost of sale, treatment, or reduction.

Tallon *v.* Vindicator Consolidated Gold Mining Co. (Colorado), 149 Pacific, 108, p. 116, June, 1915.

RELATIVE VALUATION OF MINING AND OTHER PROPERTIES.

Under the statutes of Michigan (act No. 114, Public Acts of 1911), the owner of a mine or mining property that has been assessed according to the provisions of the statute for assessing mining property for taxation can not have the assessment set aside and the taxes declared invalid on the ground that his property was valued relatively at too large a sum contrasting with valuation of other classes of real property and with the valuation of personal property.

Newport Mining Co. *v.* City of Ironwood (Michigan), 152 Northwestern, 1088, p. 1094, June, 1915.

INVALID ASSESSMENT—INJUNCTION.

An assessment of mining property for taxation is not fraudulent merely because of being excessive if the assessors have not acted from improper motive; but if purposely made too high through prejudice or a reckless disregard of duty in opposition to what must necessarily be the judgment of all competent persons, then a case is made for the equitable remedy of injunction, as the tax in such case is necessarily invalid.

Sunday Lake Iron Co. *v.* Wakefield Township (Michigan), 153 Northwestern, 14, p. 16, June, 1915.

METHODS OF ASSESSING MINES AND MINING PROPERTIES.

The statute of Michigan (Act No. 114, Public Acts of 1911), makes it the duty of the State tax commissioner to investigate, examine into, inventory, and appraise all mining properties in the State and all mineral rights which are subject to taxation and gives the commissioners authority to inspect the books and papers of any person, firm, or corporation owning, operating, or interested in mining properties or mineral rights, and also to examine under oath officers and agents of any such person, firm, or corporation, and to employ the assistance of expert engineers, and directs that the quantity and value of minerals, when known to be available therein, must be considered by the assessor in determining the value of land for taxation; and while the availability and value of minerals, unmined, are not matters of common knowledge, nor to be correctly ascertained or estimated, except by men possessed both of certain particular information and of expert knowledge, yet a number of factors must be considered in determining whether, it being apparent that there is a deposit of ore in a given locality, it is available where no mine or mining is carried on; and in such case its availability, meaning its commercial value and the ease and cost of obtaining it, must be considered as well as its quality and quantity, and accordingly if the extent and quantity of the ore body is known or estimated one important factor in determining taxable value or cash value is secured. Another factor is the price to be paid for the mining and usually this price would be a fixed price per ton during the continuance of mining operations, or a variable price, depending upon the quantity or selling price, of ore mined and shipped or both; and to obtain this price the expense of opening a mine and conducting mining operations would be incurred, and if the adventure was successful the owner of the ore mined must be paid his royalties, his selling price of the ore; but for purposes of taxation the State is not bound to accept the amount of royalty bargained for by the owner of the land and mineral as controlling its valuation, as it not infrequently occurs that where the ore body is once developed the lessee may be able to sell it upon a royalty basis in excess of the royalty provided in the lease; but in any event the miner pays the royalty, and if successful in his operations makes a profit on the ore mined. But many of these difficulties are eliminated where a mine is opened and in operation and where the quality of ore s assured and the quantity to a certain point is determinable and measurable, and where with the equipment a definite quantity of ore can be and has been mined and marketed annually and the cost of mining and the market price of the ore thus definitely fixed, and where with such equipment and operation at a given rate of mining the known ore body would be exhausted in a determinable period and during which the value of the ore would be recovered in installments. The remaining difficulty, for the purpose of assessment, is the quantity of ore or mineral not in sight and the present value of the land. If under such circumstances a rule or method exists by which mining engineers and business men ascertain the value of ore bodies for the purpose of buying and selling them, and if no better rule is or can be suggested, there is no reason why the State, for the purposes of taxation, may not use the methods of business to ascertain such values, and in thus doing the State treats the peculiar subject of taxation as the subject requires, and does not change or modify a cardinal rule of taxation, but only applies it; and the legislature has provided as an aid to assessing officers expert judgment upon the subject and the fact that experts and business men do estimate and deal in mining properties with reference to the future cost of mining and with reference to the future price of ore as well as the estimated quantity and value of the ore body, and do not regard these estimates as too uncertain to be made the basis for present valuation of a mine as a matter of business, and accordingly the State is justified in acting upon considerations upon which business men act, so long, at least, as these considerations appear to universally affect and determine, for business purposes, for buying and selling, the value of mining properties, and where such methods appear to be scientific and the result is justified by experience.

Newport Mining Co. *v.* City of Ironwood (Michigan), 152 Northwestern, 1088, p. 1094, June, 1915.

See Sunday Lake Iron Co. *v.* Wakefield Township (Michigan), 153 Northwestern, 14, June, 1915.

VALIDITY OF ASSESSMENT—GOOD FAITH OF STATE BOARD.

Where a State board of tax commissioners in fixing the value of a mine for the purpose of taxation used the information produced for it by disinterested experts of high standing in accordance with authority granted by an act of the legislature, and where one of the commissioners who had charge of the review was familiar with the development of such mines and the ore formation, acted honestly, fairly, and in good faith upon all the information at hand and made a proper and legal assessment in fixing the value of the particular mine, it can not be said that the State board acted in reckless disregard of duty, and in opposition to what must necessarily be the judgment of all competent persons, and that the assessment under such circumstances was fraudulent and the tax void, so as to entitle the owner to injunctive relief, though the assessment may in fact be too high or too low.

Sunday Lake Iron Co. *v.* Wakefield Township (Michigan), 153 Northwestern, 14, p. 17, June, 1915.

INEQUALITY OF ASSESSMENTS.

While in the assessment of property for taxation, and particularly the assessment of mining property, equality in taxation can never be achieved, yet intentional inequality of assessment will invalidate a tax.

Newport Mining Co. *v.* City of Ironwood (Michigan), 152 Northwestern, 1088, p. 1095, June, 1915.

ERRONEOUS AND VOID ASSESSMENT—DISTINCTION.

Where a local assessor has the constitutional power to assess mining property of a mine and follows the correct rule in making an assessment, he is acting within his jurisdiction; and the fact that he erred in judgment in a proper construction of the rule adopted does not show such a lack of jurisdiction as would render the tax void, but makes it merely an error in assessment. The difference consists in the absence of authority and a mistake in its exercise, and comes under the general head of irregularity, defect, omissions, mistakes, or errors of judgment and does not render the tax prima facie void; and when a tax is erroneous or illegal for the reason that the assessment is erroneous, the mine owner has an adequate legal remedy provided by the statute and can not invoke the jurisdiction of equity to have the tax declared void.

Tallon *v.* Vindicator Consolidated Gold Mining Co. (Colorado), 149 Pacific, 108, p. 116, June, 1915.

BASIS FOR DETERMINING VALUE OF MINES.

The evident purpose of section 4983 (Civil Code of Arizona, 1913) is to afford the basis for a determination of the value of a nonproducing mine or mining claim for the purpose of taxation, just as the gross product and net proceeds in dollars and cents of a producing mine or mining claim are by other sections of the statute made the basis for a determination of the value of such producing mines or mining claims for the purposes of taxation.

Earhart *v.* Powers (Arizona), 148 Pacific, 286, p. 288, May, 1915.

CORPORATION TAX—DOING BUSINESS—WHAT CONSTITUTES.

Where a mining company leased its mines and mineral lands and sold and assigned its personal property and choses in action to a lessee corporation, and for which the stockholders were to receive a certain annual stated per cent on the amount of stock, and the corporation was to be paid a certain stated sum for the purpose of maintaining its corporate existence as landlord and lessor, and held the remainder of its mineral lands for the purpose of supplying from time to time the lessee lands and mines in lieu of lands and mines described in the lease and which may from time to time be surrendered by the lessee to the lessor, is not doing business within the meaning of the corporation tax act of August 5, 1909 (36 Stat., 112), and is not subject to an excise tax under the statute, as the corporation in such case falls within the distinction that the right of income from outside property or investments of a corporation that is otherwise engaged in business, in which event the investment income may be added to the business income in order to arrive at the measure of the tax; and the right of income from property or investments of a corporation that is not engaged in business, except the business of owing property, maintaining the investments, collecting the income, and dividing it among its stockholders, and in the former case the tax is payable, while in the latter it is not.

Cambria Steel Co. v. McCoach, 225 Federal, 278, p. 282, July, 1915.

CORPORATION NOT SUBJECT TO TAX AFTER LEASE OF PROPERTY.

Where a mining corporation, with the approval of its stockholders, leased to another corporation for a term of 999 years all its property consisting of a manufacturing plant, together with the occupied and unoccupied grounds connected therewith, its mines and quarries, and roads and ways connecting the same, and an amount in acreage of coal lands and other lands connected with its mines and works, not to exceed in the aggregate 10,000 acres, and assigned and transferred to the lessee all its cash, bills receivable, accounts, licenses, leases, contracts, agreements, judgments, mortgages, stocks and bonds, and where the lessee agreed to pay as rental a sum equal to 4 per cent upon the outstanding capital stock of the lessor, together with a further sum not exceeding $5,000 to cover the cost of the maintenance of the organization of the lessor corporation, the rentals, except such cash sum, to be paid direct to the stockholders of the leasing corporation; and where the lessor corporation has maintained its corporate existence merely that it may exist as landlord and lessor, and to this end its stockholders have annually elected a board of directors and other officers and maintained books for the transfer of its capital stock, but has received no income other than as above set forth, and has done nothing else whatsoever in its corporate capacity, and has no quick assets, cash, or bank account, such lessor corporation is not liable for a special excise tax, under the corporation tax act of August 5, 1909 (36 Stat., 112), assessed against it as a corporation having a capital stock and engaged in business; and where the lessee corporation paid the tax levied under protest and to prevent the leased property from being levied upon and sold at tax sale, it may maintain an action to recover the same.

Cambria Steel Co. *v.* McCoach, 225 Federal, 278, p. 279, July, 1915.

LICENSE TAX—PERSONS SEVERING NATURAL PRODUCTS—OIL LESSEES.

The statute of Louisiana (Act No. 209 of 1912), levying an annual license tax upon each person or corporation pursuing the business of severing natural products, including all forms of timber, turpentine, and minerals, including oil and gas, sulphur, and salt from the soil, and levying a penalty for failure to report and pay the license tax, does not impose such license tax upon the owner of land who is not engaged in the business of severing the natural products from the soil, but who has leased his land and receives a royalty from the lessee who is himself engaged in the business of severing the natural product from the soil; and where a landowner has given a gas and oil lease on his land and is not in any way connected with the management or operation of the lease, and has nothing to do with the location or marketing of the oil produced from the land and does not himself operate any drilling apparatus or machinery and who does no more than receive a royalty on the oil produced, is not subject to such license tax; but in such case the license tax must be imposed upon the lessee and operator.

State *v.* Stiles (Louisiana), 68 Southern, 947, p. 948, June, 1915.

STATUTORY LIENS.

FAILURE OF OWNER TO GIVE STATUTORY NOTICE—MINER'S KNOWLEDGE OF OPERATIONS.

The failure of the owner of a mine or mining claim to give the notice required by the statute (California statutes of 1911, p. 1318), showing that a mine owned by him was operated by an independent lessee and disclaiming liability for work on the property, can not affect the rights of a miner or operator to give him a lien on the mine, where such miner has actual knowledge that the owner is not operating the mine, as the purpose of the notice was accomplished by the actual knowledge of the miner.

Street *v.* Hazzard (California Appeals), 149 Pacific, 770, p. 772, April, 1915.

RELIANCE ON EMPLOYER—KNOWLEDGE OF OPERATION BY OPTION PURCHASER.

A miner employed to work in a mine with knowledge that his employer held an option contract for the purchase of the mine, and who looked entirely to such employer for his wages, giving no credit whatever to the owner of the mine, is no more entitled to file and enforce his lien against the mine than if he had expressly agreed to waive any claim or lien against the property or any cause of actions against the mine owner.

Street *v.* Hazzard (California Appeals), 149 Pacific, 770, p. 772, April, 1915.

MINER'S RIGHT TO LIEN.

Under the California Code of Civil Procedure, section 1183, a contractor, subcontractor, superintendent, or other person in charge of any mining or work or labor in and about a mining claim, either as lessee or under a working bond or contract thereon, shall be held to be the agent of the owner for the purpose of giving workingmen and miners a lien on the mine; but this presumption raised by the statute as to the agency of the contractor or superintendent may be rebutted; and a miner or employee performing work in such a mine or upon such a mining claim, with knowledge that the persons having charge of the mine or mining operations does not own the property and was not working the mine as the owner's agent or representative, is not entitled to a lien thereon on the theory of the employer's agency.

Street *v.* Hazzard (California Appeals), 149 Pacific, 770, p. 771, April, 1915.

PERIOD OF DURATION OF MINER'S LIEN.

Section 5110 of the Idaho Revised Code gives to every person performing labor upon or furnishing materials to be used in the construction, alteration, or repair of any mining claim, flume, or tunnel a lien upon the same for work done or materials furnished; and section 5115 provides that no such lien shall bind any structure for a longer period than six months after the claim has been filed, unless proceedings be commenced in a proper court within that time to enforce the lien, and the remedy in such case is by the statute made a part of the right created and the suit must be within the time limit or the lien ceases to exist.

Continental & Commercial Trust, etc., Bank *v.* Pacific Coast Pipe Co., 222 Federal, 781, p. 784, May, 1915.

FORECLOSURE OF LIENS ON MINING PROPERTY—RANK AND PRIORITY.

The statute of Nevada regulating liens on mines and making it the duty of the court on the foreclosure of such liens to declare the rank of each lien and the class of such liens means that in each particular suit for the foreclosure of mechanics' liens on mining property the court in the judgment must declare the rank or the order of the liens; and if a party to a suit fails to exhibit his lien, he waives his rights as to any priority and to have the rank of his claim determined and stated in the judgment.

Daly *v.* Lahontan Mines Co. (Nevada), 151 Pacific, 514, p. 516, September, 1915.

FORECLOSURE OF MECHANICS' LIENS—PARTIES.

The statute of Idaho in relation to the enforcement of mechanics' liens does not in terms prescribe who shall be made parties to an action to enforce a lien, but it necessarily means that such an action must be tried against all persons whose rights, assets, or interests are claimed to be adverse and subordinate, as this is necessary in order to include such persons.

Continental & Commercial Trust, etc., Bank *v.* Pacific Coast Pipe Co., 222 Federal, 781, p. 788, May, 1915.

DAMAGES FOR INJURIES TO MINERS.

ELEMENTS OF DAMAGE.

SPECIAL DAMAGES SHOULD BE PLEADED.

In an action by an injured miner for damages, special damages by way of compensation for lost time, or expenses incurred in effecting a cure should be specially pleaded, and in such case the recovery for such items is limited to the amount stated in the pleading, and it is reversible error for a court to instruct a jury that they may allow compensation for the lost time when no claim for lost time is made in the petition.

McHenry Coal Co. *v.* Taylor (Kentucky), 176 Southwestern, 976, p. 977, June, 1915.

LOSS OF TIME—PLEADING SPECIAL DAMAGES.

In an action by a miner for personal injuries caused by slate that fell from the roof of the entry, an instruction to the effect that the jury if they find for the plaintiff should give him such sum as will reasonably and fairly compensate him for the mental pain and anguish, if any, suffered by him, which were the direct and proximate results of his injury, and for the value of the time lost by him, if any, is erroneous, where there was no claim for damages on account of the loss of time pleaded, and where the instruction did not limit either the amount to be allowed or the time for which it should be awarded.

McHenry Coal Co. *v.* Taylor (Kentucky), 176 Southwestern, 976, June, 1915.

DOUBLE DAMAGES—LOSS OF TIME AND IMPAIRMENT OF EARNING CAPACITY.

In an action for personal injuries to a miner a jury can not award damages for time lost by reason of the injuries received and at the same time give the plaintiff for a permanent impairment of his ability to earn money; but in such case the defendant may protect himself from the probable imposition of such double damages when there is a claim made in the pleadings, and request the court to instruct the jury that the allowance for any impairment of power to earn money should begin when the allowance for lost time ended, and in the absence of such request, the court of its own motion should so advise the jury in order that double damages may not be awarded.

McHenry Coal Co. *v.* Taylor (Kentucky), 176 Southwestern, 976, p. 977, June, 1915.

SETTLEMENT AND COMPROMISE.

A miner who accepts from the mine operator a sum of money in settlement of his claim for damages by reason of personal injuries received while in the course of his employment is not entitled to recover damages in an action subsequently instituted.

Tennessee Coal, Iron & Railroad Co. *v.* Moody (Alabama), 68 Southern, 274, p. 276, April, 1915.

DAMAGES NOT EXCESSIVE—INSTANCES.

A judgment in the sum of $850 for personal injuries to a miner caused by the negligence of a trip driver will not be reversed because of an erroneous instruction on the subject of punitive damages, where the evidence is convincing that the damages allowed were no more than sufficient to compensate the injured miner for the actual injuries sustained.

St. Bernard Min. Co. *v.* Ashby (Kentucky), 175 Southwestern, 626, p. 627, April, 1915.

A judgment for $800 for a miner seriously injured by an explosion of gas is not to be regarded as excessive though the miner was guilty of contributory negligence.

Oplotnik *v.* Cherokee & Pittsburg Coal & Mining Co., 148 Pacific, 616, May, 1915.

A judgment for $3,000 in favor of a miner injured by an explosion of gas is not excessive where the evidence showed that the plaintiff's sight and hearing were permanently affected.

Hartman *v.* Dickinson (Kansas), 148 Pacific, 743, May, 1915.

Where a boy fifteen and one-half years old was severely injured by being burned, his pain intense, and he was permanently disfigured and probably will never entirely recover, a court can not say that a verdict for $12,000 is excessive.

Murphy *v.* Ludowici Gas & Oil Co. (Kansas), 150 Pacific, 581, 584, July, 1915.

WATER RIGHTS.

RIGHT OF TUNNEL OWNER TO APPROPRIATE WATER.

The owner of a tunnel constructed for mining purposes and carrying water for such purposes can not maintain an action to quiet title to water flowing from a tunnel constructed by him where the evidence shows that a considerable portion of the water encountered and collected in the driving of the tunnel found its way from the openings, fissures, crevices, and seams in the rock into a creek before it was distributed or interfered with in its underground course or flow by the driving of the tunnel; and the question of whether its supply or precipitation within the surface area of a particular watershed is of no consequence; and if the tunnel owner drove his tunnel into a mountain or watershed drained by a stream, the waters of which have been appropriated and put to a beneficial use by others, and immediately under or in close proximity to such stream collects water which he claims to be "developed water," he must make satisfactory proof that such water is in fact "developed water," and it is immaterial whether the water when encountered is flowing in well defined subterranean channels or is percolating through the soil, gravel, and the fissures and crevices of the rock, as in either event the presumption is, until overcome, that the water is contributory to the main stream, and that the right to its use is vested in the prior appropriator of the stream.

Mountain Lake Mining Co. *v.* Midway Irrigation Co. (Utah), 149 Pacific, 929, 933, June, 1915.

VESTED RIGHTS PROTECTED.

A corporation organized for the purpose of ditching and conveying water for mining and other purposes over Government lands before their sale to private persons will be protected in its vested possessory title under the act of Congress of 1866 (14 Stat., 253), and such possessory title requires no further or other record title in support of the vested right of the corporation.

Happy Valley Land & Water Co. *v.* Nelson (California), 147 Pacific, 966, April, 1915.

INTERSTATE COMMERCE.

FAILURE OF CARRIER TO SUPPLY COAL CARS—JURISDICTION OF COURT.

A railroad company doing business as an interstate carrier is liable for damages caused by its failure to furnish a coal miner and operator with cars in which to load coal for shipment to points within and without the State, where it is charged that the railroad company unjustly discriminated against the coal operator in failing to distribute cars in accordance with its own rule applicable in times of shortage. The act to regulate commerce (24 U. S. Stats. at L., 379; 34 U. S. Stats. at L., 584) does not give shippers any new right but preserves existing causes of action and does not supersede the jurisdiction of State courts in any case where the decision does not involve the determination of matters calling for the exercise of the administrative power and discretion of the Interstate Commerce Commission or relate to a subject as to which the jurisdiction of the Federal courts has otherwise been made exclusive; and in actions against railroad companies for unjust discrimination in interstate commere where the rule of distribution itself is attacked as unfair or discriminatory, a question is raised which calls for the exercise of the authority of the Interstate Commerce Commission; but if the action is based upon a violation or discriminatory enforcement of the carrier's own rule for car distribution, no administrative question is involved, and such an action, although brought against an interstate carrier for damages arising in interstate commerce, may be prosecuted either in a State or the Federal courts, and the action may be based either on the common law rule or on a State statute.

Illinois Central Railroad Co. *v.* Mulberry Hill Coal Co., 238 U. S., 275, p. 282, June, 1915.

See Pennsylvania Railroad Co. *v.* Puritan Coal Mining Co., 237 U. S., 121, p. 127, April, 1915.

DUTY OF CARRIER TO FURNISH COAL CARS—LIABILITY UNDER CARRIER'S RULE FOR DISTRIBUTION.

Ordinarily a coal operator and shipper on reasonable demand is entitled to all the cars which he can promptly load with coal to be transported over the carrier's line; but this is not an absolute right, and a carrier is not liable if its failure to furnish cars was the result of sudden and great demand, which it had no reason to apprehend would be made and which it could not reasonably have been expected to meet in full, as under the common law a carrier was not required to receive all goods and passengers "if his coach be full," and the same rule applies to transportation in freight cars drawn by steam locomotives, and the law exacts only what is reasonable from such carriers, but requires that carriers should be equally reasonable in the treatment of their patrons; and in case of car shortage occasioned by unexpected demands, carriers are bound to treat shippers fairly if not identically; but in determining how an inadequate supply shall be distributed, and especially in the distribution of cars to coal companies, it may be necessary to determine whether account should be taken of system cars, foreign cars, private cars, and the company's own coal cars; but these considerations are not involved where a shipper complains that he was damaged by reason of the carrier's failure to furnish cars for the shipment of coal and did not receive the number to which it was entitled under the carrier's own rule, and that damages were sustained because of the failure of the carrier to supply cars under its own rule of distribution in case of shortage.

Pennsylvania Railroad Co. *v.* Puritan Coal Co., 237 U. S., 121, p. 133, April, 1915.

SHIPPING RATE ON CARGO COAL.

Under the Interstate Commerce Commission tariffs a certain fixed rate was placed on cargo coal from West Virginia to Toledo when for lake shipment beyond and a different tariff rate was fixed on cargo coal for Toledo only. Under these rates a cargo of coal intended for shipment beyond Toledo but which was in fact sold and delivered to lake vessels at Toledo as bunker coal, is not entitled to the tariff rate for cargo coal for shipment beyond Toledo, but is subject to the proportional rate for cargo coal to Toledo only. The reasonableness of these rates is a question for the commission and not for the court, and while the rate stands in the published tariffs of the carrier it is the only legal rate and is binding on shippers and carriers alike.

Hocking Valley Railroad Co. *v.* Lackawanna Coal & Lumber Co., 224 Federal, 930, p. 931, May, 1915.

PROPORTIONAL RATES.

A proportional rate is a part of a through rate and is the share of the aggregate charge from origin to destination for the shipment of coal which one or more of the carriers accepts for performing a definite portion of the whole transportation service, and the Interstate Commerce Commission recognizes the propriety and lawfulness of proportional rates to a point of transfer which are less than local rates to such points.

Hocking Valley Railway Co. *v.* Lackawanna Coal & Lumber Co., 224 Federal, 930, p. 931, May, 1915.

MONOPOLY—RAILROAD COMPANY ENJOINED FROM OPERATING COAL COMPANY.

The United States, under the commodity clause of the Hepburn Act (34 U. S. Stats. at L., 585), may enjoin a railroad company and a coal company from operating a coal mine and shipping the coal where it appears that the railroad company occupies the dual and inconsistent position of public carrier and private shipper and where the evidence is sufficient to show that the railroad company originally owned and operated the coal mines, for a pretended compliance with the Hepburn Act caused to be organized, through its own stockholders and officers, the coal company, and entered into a contract—restrictive contract—with its creature, neither reciprocal nor mutual, and which, in effect, prohibited the coal company from competing with the railroad company for the purchase of coal mined on the railroad lines, and considering the financial strength of the railroad company, its control of the means of transportation, its power to fix the time when transportation of coal purchased from the contracting company was to begin, its power in furnishing cars to favor those from whom it bought or to whom it sold, and the contract restraining the coal company from buying from anyone else, the contract permitting the railroad company to control or influence the price of coal, is sufficient to show a contract in restrain of trade and not a sufficient compliance with the commodity clause of the Hepburn Act as to show an absolute disassociation of the carrier from the coal company before the transportation should begin and does not leave the buyer absolutely free to dispose of and have absolute control of the coal.

United States *v.* Delaware, Lackawanna & Western Co., 238 U. S., 516, p. 525, June, 1915.

PUBLICATIONS ON MINING LAWS AND METHODS OF MINING.

Limited editions of the following Bureau of Mines publications are temporarily available for free distribution. Requests for all publications can not be granted, and applicants should select only those publications that are of especial interest to them. All requests for publications should be addressed to the Director, Bureau of Mines, Washington, D. C.

BULLETIN 17. A primer on explosives for coal miners, by C. E. Munroe and Clarence Hall. 61 pp., 10 pls., 12 figs. Reprint of United States Geological Survey Bulletin 423.

BULLETIN 45. Sand available for filling mine workings in the northern anthracite coal basin of Pennsylvania, by N. H. Darton. 1913. 33 pp., 8 pls., 5 figs.

BULLETIN 48. The selection of explosives used in engineering and mining operations, by Clarence Hall and S. P. Howell. 1913. 50 pp., 3 pls., 7 figs.

BULLETIN 53. Mining and treatment of feldspar and kaolin in the southern Appalachian region, by A. S. Watts. 1913. 170 pp., 16 pls., 12 figs.

BULLETIN 60. Hydraulic mine filling; its use in the Pennsylvania anthracite fields; a preliminary report, by Charles Enzian. 1913. 77 pp., 3 pls., 12 figs.

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BULLETIN 94. United States Mining Statutes Annotated, by J. W. Thompson. Two volumes, 1,875 pages, bound in cloth, price $2.50. Containing all United States statutes relating to mines and minerals, annotated with abstracts of all decisions construing the same.

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DEPARTMENT OF THE INTERIOR
FRANKLIN K. LANE, SECRETARY
BUREAU OF MINES
VAN. H. MANNING, DIRECTOR

MANUFACTURE OF GASOLINE AND BENZENE-TOLUENE FROM PETROLEUM AND OTHER HYDROCARBONS

BY

W. F. RITTMAN, C. B. DUTTON, AND E. W. DEAN

WITH A BIBLIOGRAPHY COMPILED BY

M. S. HOWARD

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PREFACE.

In the course of its investigations conducted for the increase of efficiency in the mineral industries the Bureau of Mines has given especial attention to the prevention of wastes in the production and utilization of petroleum. It has long been realized by the most intelligent and far-sighted producers that the insidious unseen wastes are far more serious than the visible wastes and that preventive measures must be taken if our petroleum resources are not to be prematurely depleted.

The losses, or wastes, incident to the petroleum industry may be broadly divided into two classes, as follows: (1) Losses incident to production and (2) losses through inefficient utilization.

Among the noteworthy wastes falling within the first class may be mentioned the unrecoverable oil, estimated to be more than 50 per cent of the oil in the sand. In addition there are the invisible wastes due to the inefficient protection of oil and gas bearing strata against infiltrating waters.

Losses in utilization include not only the actual waste in the use of petroleum and its products, but the making of excessive amounts of less desirable products in manufacturing the products that are in most demand. For instance, gasoline is obtained chiefly from petroleums containing a large proportion of paraffin hydrocarbons, but many of these petroleums yield only small percentages of gasoline under the old distillation methods, so that their utilization in this way has been uneconomical and wasteful. By "cracking" processes the yield of gasoline has been largely increased, but the yield should be still larger. It is estimated that the output of gasoline by the Burton process, the cracking process used by the Standard Oil companies, amounted in 1915 to 3,000,000 barrels, but this was equivalent to 18,000,000 barrels of Mid-Continent crude oil. In other words, only about one-sixth of the bulk of an average paraffin-base petroleum from the Mid-Continent field was converted into gasoline by the most efficient cracking process in commercial use in 1915.

Some striking facts regarding present conditions in the petroleum industry with especial reference to the increased consumption and existing high price of gasoline are given in a report made by the Secretary of the Interior to the United States Senate under date of February 2, 1916, and published as Senate Document No. 310.

The estimated production of gasoline in the United States in 1915 was 41,600,000 barrels and the amount exported in that year was 6,500,000 barrels, leaving a difference of 35,100,000 barrels for consumption in this country. These figures compare with a production of 12,900,000 barrels, exports of 1,640,000 barrels, and an available difference of 11,260,000 barrels in 1909; and a production of 6,920,000 barrels, exports of 1,640,000 barrels, and an available difference of 6,326,000 barrels in 1904.

In regard to the consumption of gasoline, the Secretary of the Interior says:

"The new uses of gasoline and kerosene (coal oil) are of comparative minor importance. The consumption of gasoline has been due to the tremendous growth of old uses.

"The principal uses of gasoline to-day are as follows:

"(*a*) Automobiles, motor boats, motor cycles, and air craft.

"(*b*) Stationary internal-combustion engines.

"(*c*) Traction and other portable units.

"(*d*) General industrial and household uses.

"None of these have yet reached a stage where the future can be accurately forecasted."

The Secretary states that the recent extraordinary rise in the price of gasoline is due to the following causes: The increased consumption of gasoline within the United States, which is estimated to have been 25 per cent greater in 1915 than in 1914; the increase in exports; the depletion of gasoline stocks due to increased domestic and export demands; the decreased production of crude oil containing a large percentage of gasoline; the increase in the price of crude oil; and financial influences.

A fact frequently forgotten is that, in spite of the enormous production of petroleum in this country, approximately 65 per cent of the world's output, our petroleum deposits are of limited extent as compared with our deposits of coal. The restricted area of prospective oil lands limits the possible number of new fields and makes imperative both the best use of our present known supply and the curtailing of unnecessary extravagance and of waste in the use of petroleum products. The use of oil as a fuel, when less economically valuable but equally available fuels, such as coal, are at hand can not be too strongly condemned. We are constantly acquiring knowledge of the nature of the constituents of petroleum and of the possibility of converting these constituents into much wanted and valuable products adapted to higher forms of industrial use.

In conjunction with its efforts toward minimizing of wastes such as are described above, the Bureau of Mines has established at Pittsburgh, Pa., a laboratory that is investigating problems relating to the economical and efficient utilization of petroleum and the conversion of heavier and less desirable oils into lighter and more valuable products. This is the highest type of conservation, and conspicuous success has already been attained in the early investigations, the results of which are being given to the public in this bulletin. For the future there is even greater promise of valuable additions to our knowledge in this field, to be obtained through work in the laboratory and, on a larger scale, through the cooperation and support of producers and consumers of petroleum products.

This bulletin deals with the cracking of petroleum and other hydrocarbons and the production thereby of gasoline, benzene, and toluene. The authors give a comprehensive review of the literature and present in much detail the results of the experiments made by Dr. W. F. Rittman, now chemical engineer of the Bureau of Mines, in the development of improved processes for manufacturing gasoline and benzene-toluene, and present some of the results achieved in working out the benzene-toluene process on a commercial scale.

Applications for patents on both processes have been filed with the intent of having the processes dedicated to the public and the patents assigned to the Secretary of the Interior as trustee for the people of the United States. On February 1, 1916, seven refineries, in six States, were installing plants for the gasoline process. Benzene and toluene were being produced in large quantities by the other process.

VAN. H. MANNING.

MANUFACTURE OF GASOLINE AND BENZENE-TOLUENE FROM PETROLEUM AND OTHER HYDROCARBONS.

By W. F. Rittman, C. B. Dutton, and E. W. Dean.

PART I.—THE CRACKING OF OILS; PRINCIPLES INVOLVED AND METHODS OF TREATING RECOVERED PRODUCTS.

NOMENCLATURE USED IN THIS REPORT.

In this report the ending *ene* has been used throughout, except in the bibliographv and in quotations from the writings of previous investigators, for all aromatic hydrocarbons, as, for instance, benzene, toluene, xylene. The ending *ol*, though largely used in technical description, is not correctly applicable to these hydrocarbons, and should be used to designate the impure mixtures of commerce, as distinguished from pure chemical compounds.

PRESENT STATUS OF PETROLEUM-REFINING INDUSTRY.

Although petroleum is widely distributed, being found in many parts of the world, and although information as to its occurrence and general characteristics seems to have existed from very early times, our knowledge of the chemical nature of this substance has been acquired largely within the past 40 years. Within this period a voluminous literature has accumulated, which records work as painstaking as has ever been undertaken in the whole field of chemistry, such as that of C. M. Warren, F. H. Storer, S. P. Sadtler, S. F. Peckham, and C. F. Mabery. Despite this fact, the commercial method of treating crude petroleum for the purpose of procuring marketable products is, in the main, essentially empirical. Much knowledge has been acquired as to methods of separating from crude petroleum such products as are desirable commercially, but little has been done to attain the maximum recovery of such products.

INCREASE IN PRODUCTION AND CONSUMPTION OF PETROLEUM PRODUCTS.

It is well known that the important chemical constituents of petroleum are carbon and hydrogen, with a small percentage of oxygen, and widely different proportions of nitrogen and sulphur. The many combinations and forms in which these elements exist in the various crude petroleums recovered from the earth present a wide range of problems for the chemist and the practical refiner. This statement has become particularly true in the past few years because, in spite of the enormous increase in the production of crude petroleum, not alone in the United States, but in the entire world, consumption has kept pace with supply and is continually threatening to outstrip it. Notwithstanding the jump in production in the United States from 2,000 to nearly 300,000,000 barrels of crude petroleum a year in the short space of 55 years (1859 to 1914), the diversified uses to which the many readily recoverable products have been put has called for ever-increasing supplies. The stocks of crude oil in storage above ground, notwithstanding the tremendous floods of oil that such fields as the Cushing pool, with a past maximum production of over 300,000 barrels a day, and the famous Maricopa field have produced, have remained practically constant for several years past at approximately 120,000,000 barrels. Large as this supply may seem, it represents less than six months' consumption by refineries.[a]

Evidently the race between production and consumption is of such a character that consumption will inevitably overtake production, because of the depletion of our known reservoirs of oil and the limited extent of prospective oil lands in this country.

DRAINS ON PETROLEUM OF THE UNITED STATES.

The demand for petroleum products, having been created, must continue to be supplied, and the United States, with its production of approximately 65 per cent of the world's supply, is to be drawn on in increasing measure by other countries that have learned the value and usefulness of these products and have come to be dependent on them. The world's demands must be supplied by relatively few fields, as, notwithstanding the fact that oil is found in almost every quarter of the globe, three countries—the United States, Russia, and Mexico—produce nearly 90 per cent of the world's output.

The period of 55 years that has witnessed the great increase in output of crude oils in the United States has seen also an equally great change in the relative desirability of various petroleum products. Beginning with the discovery of Prof. Benjamin Silliman, jr., of Yale University, that a satisfactory burning or illuminating oil could be obtained from crude petroleum, early efforts were devoted to the production of increasing quantities of this product. Enormous outputs were sold in the United States, and the markets were extended to foreign countries. During the year 1913, over 1,000,000,000 gallons of burning oils was exported, representing a value, in round figures, of $72,000,000. In the same year nearly $150,000,000 worth of other petroleum products was shipped to foreign lands.[a]

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[a] Day, D. T., The production of petroleum in 1913: Mineral Resources U. S. for 1913, U. S. Geol. Survey, 1914, p. 946.

PRESENT DEMAND FOR GASOLINE COMPARED WITH THAT FOR BURNING OILS.

The demand for motor fuel is outstripping that for illuminating oils in this country, and the increasing number of motor vehicles in operation, both at home and abroad, has brought about a need which, if it continues to increase at the present rate, will soon far exceed present production by ordinary means. So serious does this situation appear that recently the International Association of Recognized Automobile Clubs offered a prize of $100,000 for a gasoline substitute to cost less than gasoline. The acuteness of the need responsible for an offer of this nature becomes all the more evident when it is realized that a large proportion of the world's output of crude petroleum is of such a character as to produce by ordinary refining comparatively little gasoline and naphtha. Mexican oils are of this class. The result is that the demand for gasoline and naphtha must be supplied by the types of crude petroleums generally known as "refinable crudes." Even refinable crude petroleums are capable of yielding, by the usual refinery methods of distillation, only fractions of their total volumes as gasolines and naphthas. For example, a high-grade, refinable crude yields only between 15 and 25 per cent of refined naphthas and gasoline.

BURDEN OF SUPPLYING WORLD-WIDE DEMAND FOR GASOLINE FALLS ON UNITED STATES.

Although the United States now produces approximately 65 per cent of the world's supply of crude oils, it produces a much higher percentage of the total world output of refinable crudes. The result is that, unless efficient cracking processes are developed, the greater part of the burden of supplying the quantities of motor fuel demanded throughout the world must fall on the United States, as its chief competitors in the producing field, Russia and Mexico, furnish only relatively small quantities of such fuel.

The extent to which this demand is being met can be realized from a consideration of the exports of naphthas and gasolines during the past few years. In the period from 1910 to 1915 these exports more than trebled. During the fiscal year 1910, 77,650,923 gallons was exported; in 1911, 111,998,260 gallons; in 1912, 171,040,150 gallons; in 1914, 192,452,267 gallons,[b] and during the fiscal year ended June 30, 1915, 244,546,872 gallons.[c]

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[a] Day, D. T., Op. cit., pp. 1084–1085.

[b] Foreign commerce and navigation of United States, 1914: U. S. Bureau of Foreign and Domestic Commerce, 1915, pp. 605–607.

[c] Monthly summaries of foreign commerce of United States, January and June, 1915: U. S. Bureau of Foreign and Domestic Commerce, 1915.

The tremendous drain caused by the demand for motor fuels can be realized also by consideration of the increase in the number of motor vehicles used in this country. Five years ago that number was estimated at approximately 500,000, whereas now more than 2,000,000 motor vehicles are said to be in actual operation. If the present rate of increase continues, it will be only two or three years before this number will be doubled.

The figures cited do not include the large number of internal-combustion engines in industrial operation that use gasoline or naphtha as fuel. If it be assumed that the average power of these motor-propelled vehicles is 25 horsepower, their aggregate capacity is 50,000,000 horsepower. The significance of this figure can be conceived from the fact that the total primary power developed in the manufacturing industries amounts to less than 20,000,000 horsepower, according to the United States Bureau of the Census.[c] On the Continent, benzene obtained from the destructive distillation of coal is largely used as a motor fuel, but the percentage recovered per ton of coal is at present so small that it does not alter appreciably the world's dependence on petroleum as the chief source of supply.

TEMPORARY NATURE OF RELIEF AFFORDED BY NEW OIL FIELDS.

From time to time the bringing in of a prolific oil field relieves the strain by the great outflow of refinable oil that follows, but the relief is only temporary, as the life of a gusher field is generally short. In 1914 the Cushing pool in Oklahoma was considered a menace to the oil producers, owing to its tremendous production and the consequent decrease in price of crude oil at the well that it brought about, but by November, 1915, the prices of crude oils had attained nearly their former high levels, and producers and refiners were looking for new sources of supply. The rising market price for gasolines is the concrete evidence of present conditions. Thus, the discovery of new fields, which is daily becoming more difficult, can hardly furnish a solution of the problem of how to meet the ever-increasing demand for one of the products of crude petroleum.

Still another factor demands consideration. Even if crude oil could be obtained in sufficient quantity to meet the needs of the gasoline market an excess supply of less desirable products would be bound to result.

EFFORTS TO INCREASE YIELDS OF MOTOR FUELS.

The fact that such a large proportion of the world's supply of crude petroleum seemed to be unsuited to the production of motor fuels has led a number of investigators to undertake to work out commercially practicable processes whereby yields of these products from a given quantity of refinable oil can be largely increased, or a nonrefinable oil be made to yield the desired products. It is obvious that if such processes can be successfully developed, a better effect will result than if the production of crude oil is proportionately increased. The supply of crude oil will be conserved, and the market will not be burdened with products of limited commercial use or unprofitable to produce. These processes, of which a number have been brought forward during recent years, involve the "cracking" or decomposition of heavy oils—either low-grade natural crudes or the heavier fractions of refinable crudes—that have at present a low commercial value.

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[c] Thirteenth Census of the United States, vol. 8, Manufactures, 1909: U. S. Bureau of the Census, 1913, p. 330.

ORIGIN AND DEVELOPMENT OF CRACKING PROCESS.

The cracking process, so-called, may be said to date from its accidental discovery at Newark, N. J., in the year 1861. Essentially it consists in heating oils to such a temperature that their heavy molecules decompose into lighter molecules with the production of mixtures of lower boiling point.

FIRST APPLICATION OF CRACKING PROCESS.

As originally employed, the process was directed toward the conversion of that fraction of crude petroleum intermediate between the burning (kerosene) oils and the lubricating oils into liquid hydrocarbon products of lower density and boiling point, which would be suitable for use as illuminants. An open still was used, so that partial condensation of the vapors could occur in the upper part of the still, permitting more heat to be supplied to the boiling liquid, with consequent increase in its temperature and the desired decomposition into products of lower boiling point.

CHEMICAL NATURE OF CRACKING PROCESS.

The cracking process is simply one instance of the well-known tendency of organic substances to decompose on the application of heat. The heavier the hydrocarbon molecule, the more easily it is dissociated. In actual fact, cracking to a greater or less degree is the rule rather than the exception in the majority of distillation processes involving more than moderately low temperatures. In 1868 it was pointed out that, during the distillation of hydrocarbons on a commercial scale, cracking always takes place to some degree.[a]

A natural hydrocarbon oil, or crude petroleum, is a mixture of substances of varying boiling points. Each of the hydrocarbons of high boiling point contained in such mixtures is, during the process of distillation, exposed to various degrees of temperature below its own boiling point, so long as the hydrocarbons of lighter gravity and boiling point are not removed from the still. Consequently, the liquid hydrocarbons of high boiling point may remain exposed in the still to temperatures below their boiling point for a number of hours. For example, in refinery practice, one to three days is necessary to complete a crude distillation, the exact period depending on the size of the still and the amount of hydrocarbons to be treated. This long exposure to a degree of heat below the boiling point of a particular hydrocarbon, resulting in its dissociation or partial alteration, constitutes "cracking." As time is an important factor in every organic reaction, the technical significance of the time the oil is exposed to heat becomes evident.

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[a] Hirsch, Joseph, On the distillation of hydrocarbons: Chem. News, vol. 18, July 31, 1868, pp. 51–52.

It has been pointed out by Engler and Höfer [a] that the separation of hydrocarbons by distillation is often difficult for fractions of petroleum boiling as low as 200° C. on account of the ease of decomposition of the higher-boiling fractions at ordinary atmospheric pressures. Mabery, Zaloziecki, and other investigators have amply established this fact. In some distillation work done by two of the authors [b] it was found that a Pennsylvania oil of the best quality began to crack at 325° C. A similar figure is recorded as beginning the cracking range for Pennsylvania crudes distilled in commercial stills.[c]

FACTORS CONTROLLING CRACKING.

In the cracking of heavy oils two factors govern largely the course of the reactions that take place, namely, temperature and pressure. The function of increased temperature is to break the bonds of groups that make up the complex hydrocarbon molecule. In many processes pressure is of chief importance in controlling the temperature of distillation, but it also exerts an influence on the nature of the decompositions produced.

The temperature at which dissociation takes place with the desired rapidity is usually above the boiling point of the hydrocarbons concerned. When no pressure is employed these hydrocarbons will vaporize and pass out of the reacting sphere, the degree of alteration being small. If, however, sufficient pressure is employed to raise the boiling point to a temperature causing more rapid cracking, alteration into desired products may be obtained with a minimum of total decomposition. As soon as the action begins to be total decomposition, permanent gases will be evolved and both carbon and heavy asphaltic bodies will remain as residues. The volume of gas, therefore, if measured, is an approximate index of the degree of decomposition that is taking place.

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[a] Engler, C., and Höfer, Hans, Das Erdöl, vol. 1, 1913, p. 495.

[b] Rittman, W. F., and Dean, E. W., The analytical distillation of petroleum: Jour. Ind. and Eng. Chem., vol. 7, September, 1915, p. 754.

[c] Robinson, F. C., The manufacture of petroleum products: Met. and Chem. Eng., vol. 11, July, 1913, p. 389.

OBJECT OF CRACKING PROCESS.

The cracking process involves, therefore, a partial alteration or dissociation, as distinguished from a more complete alteration or decomposition. The object of cracking is to alter the heavier molecules so that low-boiling hydrocarbons will be produced that would not appear on ordinary distillation. Such a process may or may not be accompanied by an appreciable production of permanent gases, depending on the degree of alteration necessary to obtain the desired products. In a simple cracking process as high a temperature as is practicable within the limits of the apparatus is desirable, in order to insure a high speed of reaction. A decrease in temperature below a given point will mean a decided increase in time. In commercial practice this would mean increase in operating costs. On the other hand, too high a temperature will mean a large production of extreme decomposition products, so that for results desirable commercially a mean temperature must be chosen that will cause the process to be completed at the maximum speed consistent with a relatively small production of permanent gases and carbon. This consideration has been emphasized by Engler and Höfer, [a] who point out that, for the splitting of heavier petroleum hydrocarbons into lighter products, excessively high temperatures are not advantageous.

COMMERCIAL VALUE OF CRACKING PROCESS.

Liquid-phase cracking processes, though extensively used for the production of illuminating oils and motor fuels, are as yet in the primary stages of commercial application. As a means of supplying the products most in demand, their importance can hardly be overestimated. They served their purpose well for producing burning oils, when these were the most desirable products, and are now being adapted to the production of gasolines and naphthas. It only remains to discover the most efficient means of obtaining the maximum possible percentage of the desired product from any particular oil. The ease and cheapness with which crude petroleum can be transported to regions lacking supplies of crude oil give particular value to cracking processes, as the cheapest and most readily obtainable oils may be subjected to cracking at the place of the greatest demand and most extensive use.

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[a] Engler, C., and Höfer, Hans, Op. cit., vol. 1, p. 565.

PREJUDICE AGAINST PRODUCTS OF CRACKING PROCESSES.

There has been considerable prejudice against the products of cracking processes. It has been claimed that illuminating oil made by cracking is inferior in light-giving power and more easily acquires a dark color with age. The chief objection seems to have been due to the inevitable presence of unsaturated hydrocarbon compounds. These have an unpleasant odor and require the use of a large quantity of sulphuric acid in the refining process, which increases the cost of the finished commercial product.

The same prejudice seems to exist with respect to the cracked gasolines that have been put on the market. The characteristic odor of the cracked products is attributed to overheating or overcracking of some of the constituents, a trouble that is avoided with difficulty in treating a complex mixture of substances of widely different boiling points. Only a small part need be overheated to impart a characteristic odor to the whole mixture. However, refining with sulphuric acid eliminates a great deal of this odor.

The volume of unsaturated hydrocarbons, or olefins, produced has been one of the chief drawbacks to all the cracking processes operated at atmospheric pressure. The well-known fact that unsaturated hydrocarbons tend to form saturated hydrocarbons by the action of heat with pressure is largely responsible for the number of processes that have been brought forward for cracking under pressure, more particular mention of which is made later. For the same reason many inventors introduce catalyzers into the cracking zone.

Another objection made to the cracked gasolines has been the alleged fact that such gasolines deposit larger quantities of soot in the cylinders of internal-combustion engines than do uncracked gasoline distillates. It is conceivable that such a deposit may be largely due to improper combustion, which occurs because the nature of the product is unknown. Hall,[a] who has investigated this subject, has stated that such soot deposition is attributable to this cause, the mixture used being too rich. His investigations tend to show that a properly cracked gasoline or motor spirit gives a greater mileage than a gasoline distillate, which he explains on the ground that the cracked gasoline burns more slowly than the "straight" distillate, thereby imparting power during the entire stroke of the piston, whereas the distillate gives off its power during only a fraction of the stroke. The observations of Hall have been confirmed by the experience of others.

Therefore there is reason to believe that instead of being inferior the cracked gasolines are really superior for use in automobile engines, as regards both mileage and power obtainable, even though they lack some desirable properties, and this suggests that a blend of cracked gasolines and the "straight" distillate gasolines may give greatly improved results. The Pittsburgh laboratory of the Bureau of Mines is now working on this problem.

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[a] Hall, W. A., Address delivered to Institution of Petroleum Technologists, London, Feb. 18, 1915; abstracted in Petroleum World, vol. 12, March, 1915, pp. 137-145.

HISTORICAL REVIEW OF PATENTED CRACKING PROCESSES.

FIRST PROCESSES.

The first processes for the recovery of lighter boiling hydrocarbons from heavier hydrocarbons were used, as has been previously mentioned, for the production of burning oils. The first patent of this character found by the authors was that granted to Young in 1865.[a] The distillation was conducted in a closed vessel provided with a loaded valve, which was set so that the vapors would escape at any desired pressure. A pressure of 10 to 20 pounds per square inch is specified, and the patent seems to have been directed to the recovery of burning oils from Scottish shale oils.

One of the earliest patents taken out in this country for increasing the yields of kerosene or burning oils was that granted to Benton.[b] In Benton's process the liquid oil was conducted through a series of pipes inclosed in a furnace, where the temperature of the oil was raised to 371° C. (700° F.) to 538° C. (1,000° F.), under a pressure of 500 pounds per square inch, or less. The heated oil was then expanded into a chamber kept at approximately the same temperature, and was conducted thence to a condenser.

In the same year (1886) a patent was granted to A. Riebeck's Mining Works,[c] Inc., in Halle, for a process involving distillation under pressure of heavier oils to produce benzine and burning oils. A pressure-release valve was placed between the still and the condensers. When the pressure on the still exceeded that for which the valve was set, the valve automatically opened and allowed the vapors to pass to the condenser.

These processes were followed by the process patented by Dewar and Redwood.[d] This process eliminated the valve between the still and the condenser and permitted the two to be in free communication, the space above the liquid in the still and condenser being charged with air, carbon dioxide, or other gas under the required pressure. The condenser was provided with a regulated outlet for the liquid products.

A somewhat later patent with the same object, the production of burning oils, etc., is that of Ragosin,[e] which seeks to avoid the use of pressure by introducing into the still a hydrocarbon of low boiling point, such as benzene, the combined vapors of the benzene and initial material passing to a superimposed cracking vessel, placed in the same furnace construction but with separate means of heating. The liquids that form in this cracking vessel run back into the still. The cracked vapors are passed through a series of fractional condensers arranged in towers, the vapors entering at the bottom and ascending through the series.

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[a] Young, James, English Patent 3345, Dec. 27, 1865.

[b] Benton, G. L., U. S. Patents 342564 and 342565, May 25, 1886.

[c] A. Riebeck's Mining Works, Inc., in Halle, German Patent 37728, Klasse 23, Feb. 28, 1886.

[d] Dewar, James, and Redwood, Boverton, English Patents 10277, June 24, 1889; U. S. Patents 419931, Jan. 21, 1890; 426173; Apr. 22, 1890.

[e] Ragosin, V. I., English Patent 1411, Jan. 18, 1898.

CRACKING PROCESS FOR THE PRODUCTION OF GASOLINE.

A large number of processes have been patented for the production of increased quantities of gasoline or motor spirits from the heavier hydrocarbon mixtures by destructive distillation.

Of all these processes only one so far as known is in extensive use in the industry, this being the well-known Burton process, which is owned and controlled by the Standard Oil Co. of Indiana, and has been rather generally employed by the various Standard Oil companies at different periods. The fact that the Standard Oil marketing companies have put in large and expensive installations involving the use of this process is the best possible indication of the need of efficient cracking processes in the industry. With their usual foresight these companies have perceived that the supplies of crude petroleum would not satisfy the continually increasing demand for motor spirits. The fact that to a large degree the employment of the Burton process has improved the situation so far as these companies are concerned is a tribute to the sagacity of the companies, as well as to the intrinsic merits of the process itself. There can be no question that the use of this process in the past few years by the Standard Oil companies has contributed greatly to relieve threatened shortages in the supply of gasoline. The process itself has been a real conservation factor so far as American petroleum resources are concerned, and too much credit can not be given to the inventor as well as to the companies whose faith and energy have made its successful development possible.

CLASSES OF GASOLINE-CRACKING PROCESSES.

Broadly speaking, gasoline-cracking processes may be divided into three types—those that involve cracking at atmospheric pressure; those that involve cracking under pressure in stills and large vessels; and those that involve cracking under pressure in small vessels or tubes.

The disadvantage of processes involving cracking at atmospheric pressure is the large volume of olefins formed. It is also difficult to obtain cracking in any effective degree, as the higher temperatures can not be attained without causing vaporization, and without pressure the vapors can not be held in the heated zone. Fuel consumption is considerable as the operation has to be conducted slowly to give satisfactory results. The degree of conversion is necessarily less at atmospheric pressure than under higher pressures as, on account of the greater stability of the lighter molecules, only the heavier molecules are subject to alteration.

In the processes involving the use of pressure as an aid to cracking, some of the investigators lay emphasis on having the condensation occur under pressure, and others favor condensation after release of pressure. Burton [a] claims that by having the pressure-controlling valve placed beyond the condenser, so as to have the condensation take place under a pressure of approximately 75 pounds, the volume of unsaturated hydrocarbons is largely reduced. On the other hand, Brooks, Bacon, and others [b] describe experiments conducted first with the valve between the still and the condenser, and then with the still and the condenser in free communication so as to allow condensation under pressure, the conditions remaining otherwise the same. They report that no difference was observable in the volume of olefins formed.

One disadvantage of processes in which the vapors are permitted to escape only through a pressure-release valve, such as those patented by A Riebeck's Mining Works Inc., in Halle,[c] and others,[d] is the irregularity of the distillation. This irregularity is obviated by having condensation take place under pressure, and theoretical considerations favor such condensation.

LIMITATIONS OF CONVENTIONAL CRACKING PROCESSES.

Hall [e] and others [f] have pointed out the great danger inherent in cracking operations in which a large volume of liquid is heated under pressure in a still or other large container. As discussed more fully later, a two-phase system—that is, one in which both liquid and vapor are present—necessitates the employment of a given pressure for a given temperature, and vice versa, unless one of the phases be caused to disappear. Therefore, in order to reach the high temperatures that are desirable in order to bring about a maximum of cracking, it is necessary to use correspondingly high pressures if the two-phase system is to be maintained. This use of both high temperature and high pressure is a source of danger when large quantities of liquids are treated in a single operation, as the retainers are apt to give way and the great volume of highly heated liquid becomes a serious menace. A carbon deposit accumulates in the stills and large retorts employed in pressure-cracking processes, which on account of its high insulating power causes the vessels to burn out during prolonged runs. Recently this difficulty has been largely obviated by the use of stills with seamless bottoms.[a]

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[a] Burton, W. M., U. S. Patent 1049667, Jan. 7, 1913.

[b] Brooks, B. T., Bacon, R. F., and others, The preparation of gasoline and kerosene from heavier hydrocarbons: Jour. Ind. and Eng. Chem., vol. 7, March, 1915, p. 180.

[c] A. Riebeck's Mining Works, Inc., in Halle, German Patent 37728, Klasse 23, Feb. 28. 1886.

[d] Bacon, R. F., and Clark, C. W., U. S. Patent 1101482, June 23, 1914; Young, James, English Patent 3345, Dec. 27, 1865.

[e] Hall, W. A., Address delivered to Institution of Petroleum Technologists, London, Feb. 18, 1915; abstracted in Petroleum World, vol. 12, March 1915, p. 137.

[f] See Clark, E. M., U. S. Patent 1119496, Dec. 1, 1914; Humphries, R. E., U. S. Patent 1122022, Dec. 22, 1914.

Processes of this nature are also subject to the limitation common to all two-phase (liquid-vapor) systems, namely, that the temperature and the pressure can not be varied independently of each other. This fixed interdependence of temperature and pressure is one of the greatest handicaps of all two-phase processes, for the higher temperatures and pressures favorable for cracking the lighter and intermediate fractions of a crude petroleum can not be attained because of the difficulty in finding retainers that will withstand the necessary strain. For example, such interdependence makes impracticable the use of kerosene (burning oils) as an initial material in the production of gasoline.

CRACKING AT ATMOSPHERIC PRESSURE.

A considerable number of processes have been brought forward for effecting at atmospheric pressure the cracking of heavier hydrocarbons into gasoline hydrocarbons.

Laing[b] proposed to accomplish this result by passing the vapors coming from the still through a superheater placed in the flue connected with the oil still, a suitable arrangement being provided for regulation of the heat.

The process patented by Seigle[c] provides for the passage of the initial hydrocarbon material through a series of vaporizing chambers, and thence through a series of heating or cracking chambers where the vapors are superheated and cracked. Heat is supplied by immersing both series of chambers in a bath of molten lead.

Cowper-Coles[d] in 1906 patented a process for obtaining low-boiling hydrocarbon oils from liquid hydrocarbon distillates obtained from shale, peat, coal, etc. The initial material is heated in a still and the vapors then pass through a series of small pipes heated to a temperature of about 925° C., from which they are conducted to a condenser. The size of pipes is not indicated.

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[a] See Humphries, R. E., U. S. Patents 1122002, 1122003, Dec. 22, 1914.

[b] Laing, John, English Patents 11079, May 16, 1891; 4120, Mar. 17, 1890.

[c] Seigle, Adolphe, U. S., Patents 567751, 567752, Sept. 15, 1896; French Patents 249583, Aug. 13, 1895, 252068, Nov. 29, 1895; English Patents 18518, Oct. 3, 1895, 23235, Dec. 4, 1895.

[d] Cowper-Coles, S. O., English Patent 27945, Dec. 7, 1906.

Noad[a] passes a mixture of oil and water, in the proportion of two parts of oil to one of water, into one end of a U-shaped chamber filled with scrap iron, and maintained at a dull red heat (about 482° C.), the vapors then being led to a condenser.

A process of somewhat similar nature to that of Noad's, aside from the different construction of the converter, is that controlled by the New Oil Refining Process (Ltd.), an English corporation.[b]

An emulsion of oil and water is fed into a vertical tube, which may be constructed of fire clay or metal. This retort, which is inclosed in a furnace, is filled with scrap iron or other refractory material, and is heated to 538° to 650° C. The oil enters the top of the retort by gravity feed, and the water is injected into the bottom of the tube in a similar manner. The vapors pass upward out of the retort into a first condenser where, it is claimed, the unconverted vapors are condensed; thence they return to the retort for further treatment, and the lighter converted vapors pass into a second condenser. The oil that remains liquid at the temperature in the interior of the retort is drawn off at the bottom.

In the process patented by Adams[c] oil is fed into a converter into which an electric heater is introduced. The liquid oil is maintained under a partial vacuum and is forced upward through an insulating shell into the interior of the converter so as to localize the heating of the oils. The heater, which is maintained at a "cherry red" or or "white heat," is kept submerged to avoid explosion of the oil vapors. The vapors are then passed to a condenser.

Moeller and Woltereck[d] pass vapors of heavy oil, mixed with steam, superheated to 700° to 800° C. at atmospheric pressure, through tubes filled with broken coke and heated to a dull red heat (600° to 800° C.).

Another process involving the use of steam is that patented by Valpy and Lucas.[e] Superheated steam is introduced into a still containing liquid oil, and the mixture of steam and oil vapors resulting is passed through cracking tubes inclosed in a furnace chamber. The tubes are filled with a catalytic material (thin nickel tubes electrically heated may be used) and are heated to a temperature varying between "dull and bright red." The decomposed steam and oil vapors are then passed to a condenser. Provision is made for the mixed vapors to traverse a pair of coils, one of which is placed in the still and the other in the oil-supply tank, as a means of adding heat to the oil and cooling the vapors before they enter the condensers.

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[a] Noad, James, U. S., Patent 971468, Sept. 27, 1910.

[b] The New Oil Refining Process (Ltd.), and Neilson, R. G., English Patents 13675, 1908; 28460, Dec. 18, 1911; 20074, 20075, Aug. 28, 1913.

[c] Adams, J. H., U. S. Patent 976975, Nov. 29, 1910.

[d] Moeller, Julius, and Woltereck, H. C., English Patent 16611, July 19, 1913.

[e] Valpy, O. H., and Lucas, O. D., English Patent 20470, Sept. 10, 1913.

A more recent process invented by Greenstreet[a] proposes to atomize crude oil with steam, to pass the mixture of oil and steam through a coil of pipes maintained at a temperature above 538° C. to ignite a part of the product, and then to condense the remainder.

Sabatier,[b] one of the most noted inventors in the hydrocarbon field, has recently brought forth a new process for the production of light hydrocarbons, involving the use of catalytic material. The oil is first vaporized and the vapors directed against platinum wires rendered incandescent by electric current and brought to a bright red heat. Hydrogenation of the unsaturated hydrocarbon is accomplished by passing the vapors through a spiral of nickel or other suitable catalytic material heated to 200° to 350° C. by an electric current.

Several processes have been devised for facilitating the cracking reaction at atmospheric pressure by adding aluminum chloride to the oils to be treated.

The first process of this nature seems to have been that of Friedel and Crafts.[c] From 5 to 20 per cent of anhydrous aluminum chloride is added to the hydrocarbon material and the mixture heated in a still or other large retort to a temperature varying from 100° to 600° C. The process was designed to produce burning oils, lubricating oils, etc., from heavier hydrocarbons. Gasoline, of course, was not in demand at that time, so that the adaptability of the process for gasoline production was not mentioned, although clearly, in the light of later knowledge, the process is equally suitable for this purpose.

Gray,[d] an American inventor, has patented a similar process with the addition of a mechanical stirrer which keeps the aluminum chloride in suspension in the hydrocarbon material and prevents its being overheated. The mixture is heated to a temperature not higher than 163° to 177° C. Usually 5 to 10 per cent of anhydrous aluminum chloride or other metallic chloride is used.

The more recently patented process of McAfee[e] proposes the use of the same percentage admixture of aluminum chloride as specified by Gray, and approximately the same temperatures (149° to 177° C.). No mechanical stirrer is used. A new feature is the use of a separate chamber between the still and the first condenser, for the separation and recovery of the aluminum chloride.

Although these are by no means all the patented cracking processes, they sufficiently illustrate the development of the art for processes operated at atmospheric pressure. Other patents are mentioned in the bibliography at the end of this bulletin.

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[a] Greenstreet, C. J., U. S. Patent 1110923, Sept. 15, 1914; French Patent 446475, July 24, 1912.

[b] Sabatier, Paul, and Mailhe, Alphonse, U. S. Patent 1124333, Jan. 12, 1915.

[c] Friedel, Charles, and Crafts, J. M., English Patent 4769, Dec. 15, 1877.

[d] Gray, G. W., English Patents 17838 and 17839, Aug. 5, 1913.

[e] McAfee, A. McD., U. S. Patent 1127465, Feb. 9, 1915.

CRACKING UNDER PRESSURE.

In order to point out the various lines along which efforts have been made to develop cracking processes, as well as to more clearly differentiate from prior conceptions the processes discussed in this report, a brief review of the patents granted for processes involving cracking under pressure is given. The improvements in the art are naturally protected by patents, so that the patent literature contains the record of the most recent and important developments. The patented processes covering the utilization of cracking under pressure for the production of burning or illuminating oils have already received attention.

The danger incident to the subjection of large volumes of liquid to high temperatures and pressures has led many investigators to adopt the use of tubes in lieu of stills or similar large retorts. The most striking feature, therefore, of the development of the cracking art is the increasing number of patents involving the employment of small pipes or tubes. A number of recent investigators have called attention to the improved results obtained by condensation under pressure, among these being Hall,[a] Brooks, Bacon, and others, [b] and Humphreys. [c] All the more recent patents seem to involve the use of pressure condensation as a means of obtaining increased yields.

The best known and most successful process employing the use of a large retort or still for the cracking of heavier hydrocarbons is that patented by Burton. [d] The process is expressly stated to be limited to petroleum distillates with a boiling point of 500° F. (260° C.) or above. The initial material is placed in the still and subjected to a temperature of 650° F. (343° C.) to 800° F. (426° C.) and a pressure of 4 to 5 atmospheres. The vapors are condensed under pressure in an ordinary type of condenser.

A further proposed modification [e] of this process involves the addition of inclined conduits—pipes 12 inches in diameter and 40 feet in length—passing from the still to the condenser. The object of the inclined conduits is to permit the condensation of the heavier fractions of the distillate, which automatically return to the still for further exposure to the cracking temperature, the heavier fractions being thus subjected to the repeated action of the pressure and temperature in the still.

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[a] Hall, W. E., Address delivered to Institution of Petroleum Technologists, London, Feb. 18, 1915; abstracted in Petroleum World, vol. 12, Mar., 1915, p. 137.

[b] Brooks, B. T., Bacon, R. F., and others, The preparation of gasoline and kerosene from heavier hydrocarbons: Jour. Ind. and Eng. Chem., vol. 7, Mar., 1915, p. 180.

[c] Humphreys, R. E., U. S. Patent 1119700, Dec. 1, 1914.

[d] Burton, W. M., U. S. Patents 1049667, Jan. 7, 1913; 1105961, Aug. 4, 1914. See also Clark, E. M., U. S. Patent 1132163, Mar. 16, 1915.

[e] See Humphreys, R. E., U. S. Patent 1119700, Dec. 1, 1914.

The process patented by Leffer[a] provides for the distillation of heavy hydrocarbons in an ordinary still, the liquid oils being agitated by a mechanical stirrer. The system is under a pressure of 10 to 12 atmospheres, created by a circulating current of permanent gases. The vapors coming from the still are then passed through a decomposition chamber maintained at a temperature not exceeding 410° C., the chamber being placed between the distillation vessel and the condenser.

In a later modification of the same process Leffer[b] proposes to atomize the hydrocarbon material to be treated in a mixing chamber by means of a circulating current of permanent gases and to pass the liquid in a finely divided form into a decomposing chamber maintained at a temperature not exceeding 400° C., whence the vapors pass through a dephlegmating column to a condenser. The specification provides for atomization directly into the decomposing chamber instead of in the mixing chamber, if desired.

In the process discovered by Planes, Ltd., and Thompson[c] oil is heated in a high still, the bottom being of conical shape and tapering down to a pipe through which oil, hydrogen gas, and catalyzers are introduced. The upper half of the still is outside the furnace chamber, thereby permitting the condensation of heavy vapors. Heating takes place in the presence of finely divided nickel, the system being under a pressure of 5 to 100 pounds per square inch. Hydrogen, or a purified water gas, is forced into the still under heavy pressure and the liquid mixture is violently agitated by a mechanical stirrer. The vapors pass to the condenser through a valve loaded to insure the desired pressure and enter the condenser after passing through a series of perforated shelves which are covered with finely divided nickel deposited on asbestos.

The process devised by Bacon and Clark[d] proposes to heat hydrocarbons with boiling points of 250° C. and upward under a pressure of 100 to 300 pounds per square inch. A pressure-release valve is placed between the still and the condenser, so that condensation occurs at atmospheric pressure. Temperatures (300° to 500° C.) sufficient to permit the use of the pressures specified are employed. The process is distinguished from the Burton process by the use of higher pressures and the specification of lower boiling hydrocarbons as the starting material.

The process jointly patented by Shedlock and the Optime Motor Spirit Syndicate (Ltd.),[e] proposes to first purify the oils to be treated by removing sulphur, carbon, asphaltic bodies, etc., the oils being passed through a vessel charged with metallic oxides. This vessel is heated in a furnace to a temperature of 149° to 204° C., under a pressure of 3 to 6 atmospheres (45 to 90 pounds per square inch). The purified oils are then conducted through a settling vessel into an agitator, where the liquid oil is emulsified with water, the emulsion being subsequently forced, under a pressure of from 3 to 6 atmospheres, through a converter filled with iron or steel shavings. This converter is heated to 260° to 482° C. The emulsified products are then expanded through small jets into a large chamber, from which they are withdrawn by means of a pump and passed through a scrubbing vessel, where the heavier oils are recovered, the lighter oil vapors and permanent gases passing onward into a condenser.

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[a] Leffer, L. G., English Patent 19051, Aug. 20, 1912; French Patents 439476, Jan. 29, 1912; 16360, Aug. 19, 1912.

[b] Leffer, L. G., English Patent 4140, Feb. 18, 1913.

[c] Planes, Ltd., and Thompson, W. P., English Patent 5245, Mar. 1, 1913.

[d] Bacon, R. F., and Clark, C. W., U. S. Patent 1101482, June 23, 1914.

[e] Shedlock, J. J., and Optime Motor Spirit Syndicate (Ltd.), English Patent 1878, Jan. 23, 1914.

CRACKING IN TUBES OR PIPES.

In 1908 Testelin and Renard [a] obtained letters patent for a process involving the passage of steam and liquid oil through a system of tubes inclosed in a furnace chamber, the tubes being kept at a temperature of 400° to 450° C. The mixture is then passed through another series of tubes filled with pieces of burnt porous clay and maintained at a temperature approximating that of red heat. The feed of steam and petroleum is so regulated that equal quantities of water and petroleum are condensed.

Hall [b] has brought forward a process involving the passage of a liquid hydrocarbon under a pressure of 70 pounds per square inch through a coil of pipe heated to approximately 650° C., the vapors then passing to a cooling chamber, where the temperature is lowered to approximately 200° C., and thence to a condenser, condensation taking place under pressure. In an improved form of this process [c] the oil is vaporized and passed at a rate of feed exceeding 70 gallons per hour through winding inclined metallic tubes 1 inch in diameter and 300 feet in length, in which are inserted rods of catalytic material, preferably nickel. The products are passed from these tubes into an expansion chamber, where the pressure is reduced from that existing in the cracking tubes (about 4 atmospheres) to atmospheric pressure. The expansion chamber consists of a tube 12 inches in diameter and 12 feet high. The speed of the vapors through the tubes approximates 6,000 feet per minute. The speed is reduced by causing the vapors to enter the expansion chamber through a confined space acting as a throttle and serving to impinge them against a baffle. It is claimed that the reduction of pressure causes a higher temperature in the large tube than in the cracking tubes, so that an additional superheating of the vapors and cracking takes place. The carbon is collected at the bottom of the expansion tube upon a metal screen.

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[a] Testelin, Auguste, and Renard, Georges, English Patent 16881, Aug. 11, 1908.

[b] Hall, W. A., English Patent 24491, Oct. 28, 1913.

[c] Hall, W. A., English Patent 17121, July 25, 1913.

Kittle,[a] by means of a compression air pump, forces liquid oil under a pressure of 100 pounds per square inch through a coil of pipe three-sixteenths of an inch in diameter and at least 20 feet long. The coil is immersed in a molten-lead bath, the temperature of the bath being at least 700° C., so that the temperature of the oil in the tubes will be approximately 500° C.

Another process, in which a catalyzer is used to promote cracking, is that of Ellis,[b] in which the oil, after having been preheated, is atomized into the top of a vertical tube or chamber completely filled with catalytic material. A pressure of 30 to 60 pounds per square inch is maintained by controlling the discharge of the gases produced. A volume of air is admitted into the chamber sufficient to effect combustion of 3 to 10 per cent of the oil in order to crack or convert a part of the remaining hydrocarbon material.

In the process patented by Turner[c] a mixture of steam and liquid oil is carried through a series of coiled pipes, 2 inches in diameter, the coils being so arranged that any desired amount of superheating of the mixture of steam and oil vapor can be obtained. The products then pass through a series of fractional condensers. A temperature of 1,000° F. (538° C.) to 1,400° F. (760° C.) and a pressure of about 60 pounds per square inch are specified.

Von Groeling's[d] process also involves the use of steam. Oil from a series of tubes externally heated in a furnace is fed into a still into which steam is introduced. The mixed vapors of steam and oil pass to the top of the still and then repass through the same tubes, so that the vapors from the still are forced to travel through the current of oil proceeding to the still. In this manner the oil is preheated before it reaches the still. A temperature of 350° to 500° C. is maintained both in the still and in the cracking tubes. The vapors, after passing from the cracking tubes, enter a dephlegmator and finally a condenser, where they are liquefied without pressure.

In Zerning's[e] process water and liquid oil are forced by means of a pump through tubes heated to 300° to 400° C., the resulting vapors being rapidly passed through pipes heated to a "dark red" heat, whence they are passed through a cooling device, where the water and tarry substances are condensed. The uncondensed vapors and fixed gases are collected in a gas reservoir and then driven by means of force pumps through the hydrocarbons that are to be treated. The pressure used is not specified, but it is claimed that too high pressures should be avoided.

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[a] Kittle, H. A., English Patent 8336, Apr. 6, 1909.

[b] Ellis, Carleton, English Patent 25631, Nov. 8, 1912.

[c] Turner, C. W., U. S. Patent 1046683, Dec. 10, 1912. See also U. S. Patent 726379. Apr. 28, 1903.

[d] Von Groeling, A. F., English Patent 10213, Apr. 30, 1913.

[e] Zerning, Hermann, English Patent 731, Jan. 9, 1913.

Clark[a] has patented a process somewhat similar to that involved in the Zerning[b] patent except that water is not introduced. The liquid oil to be treated is charged into a large tank or receiver, from which the oil is withdrawn by means of a pump and circulated in a fine stream through a coil inclosed in a combustion chamber. The heated liquid oil is then returned to the receiver. By continued circulation, the oil in the receiver is raised to a temperature of 343° to 454° C., and a pressure of 3 to 7 atmospheres (45 to 105 pounds) is built up by controlling the escape of the vapors by a pressure-release valve placed beyond the condenser.

Bacon and others[c] have worked out an improvement on their earlier process[d] in which liquid oil is fed by a force pump into the top of a vertical pipe or retort 19 inches in diameter and 20 or more feet in length, inclosed in a furnace chamber. The liquid oil is maintained at a level above the top of the furnace combustion chamber, the retort or tube projecting some 5 feet above the top of the furnace. The liquid oil is heated to a temperature of 350° to 500° C., under a pressure of 60 to 300 pounds per square inch. A settling chamber is provided at the bottom of the retort into which the tar and coke formed in the cracking zone of the retort can be deposited and be withdrawn from time to time.

A more recent and novel process is that of Dubbs[e] in which oil and water are heated in coils, under a pressure of 50 to 600 pounds per square inch, to a temperature of 66° to 316° C., and the mingled vapors are then discharged through a suitable jet into an expansion chamber and the pressure released. Additional heat is admitted into the expansion chamber when necessary.

HYDROGENATION OF PRODUCTS OF CRACKING.

It will be noted that many of these inventors have attempted to combine with the cracking process that of hydrogenation, in order to minimize the formation of the unsaturated hydrocarbons characteristic of low-pressure cracking reactions. This they propose to accomplish either by the decomposition of steam, which is mingled with the oil vapors so as to produce free hydrogen, the liberated oxygen combining with free carbon to form carbon monoxide, or else by the employment of a catalyzer to break down sufficient of the hydrocarbons to provide free hydrogen for the saturation of a considerable proportion of the remaining hydrocarbons. The combined use of steam and catalyzers has been attempted, as in the processes patented by Lamplough,[a] Testelin, and Renard,[b] New Oil Refining Process (Ltd.),[c] Valpy and Lucas,[d] Noad,[e] and Shedlock and Optime Motor Spirit Syndicate (Ltd.).[f]

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[a] Clark, E. M., U. S. Patent 1119496, Dec. 1, 1914.

[b] Zerning, Hermann, English Patent 731, Jan. 9, 1913.

[c] Bacon, R. F., Brooks, B. T., and Clark, C. W., U. S. Patent 1131309, Mar. 9, 1915.

[d] Bacon, R. F., and Clark, C. W., U. S. Patent 1101482, June 23, 1914.

[e] Dubbs, J. A., U. S. Patent 1135506, Apr. 13, 1915. See also U. S. Patent 1100717, June 23, 1914.

AROMATIC HYDROCARBONS.

PREVIOUS SOURCES OF SUPPLY.

The chief sources of aromatic hydrocarbons heretofore have been coal tar, water-gas tar, and the coal gases from by-product coke-oven installations.

From these different sources of supply the aromatic hydrocarbons are recovered as by-products. The utilization of these tars dates from the discovery by Perkin in 1856 of the first anilin dye. Coal tar, which was the first source of these aromatic compounds, was once considered a great annoyance in the gas industry, yet the compounds obtainable therefrom have rendered it, next to the gas, the most valuable of all the products obtained by the distillation of coal. In fact, the president of one of the large steel companies recently told the authors that because of the condition resulting from the European war the value of by-products obtained from the coke oven installation operated by his company was 50 per cent greater than the value of the coke. In other words, the coke had temporarily become the by-product.

Although the coal tars continue to be one of the chief sources of supply, the great extent to which carbureted water gas has replaced coal gas, particularly in the United States, has brought water-gas tar into prominence as an important source of supply.

The prejudice that previously existed against the aromatic hydrocarbons derived from water-gas tar is largely disappearing as the proper method of utilizing these materials is becoming better understood. For example, it was formerly maintained that toluene from water-gas tar was not suitable for the manufacture of trinitrotoluene. The fact that to-day it is being used for this purpose in increasing quantities and that it constitutes one of the important sources of supply in the United States, shows that this objection no longer exists.

The great increase in the number of by-product coke-oven plants in operation in this country makes coal gas more important than coal tar as a potential source of supply. It has been estimated that a high-grade gas coal yields approximately 1 per cent by weight of light oil when the gas is washed. The value of this product to-day is nearly 20 per cent of that of the coal.[a] The light oil obtained from tar represents only about 0.2 per cent of the coal.

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[a] Lamplough, Frederick, English Patent 19702, Aug. 28, 1912.

[b] Testelin, Auguste, and Renard, Georges, English Patent 16881, Aug. 11, 1908.

[c] The New Oil Refining Process (Ltd.), and Neilson, R. G., English Patents 13675, June 27, 1908; 28460, Dec. 18, 1911; 20074, 20075, Sept. 3, 1912.

[d] Valpy, O. H., and Lucas, O. D., English Patent 20470, Sept. 10, 1913.

[e] Noad, James, U. S. Patent 971468, Sept. 27, 1910.

[f] Shedlock, J. J., and Optime Motor Spirit Syndicate (Ltd.), English Patent 1878, Jan. 23, 1914.

INDUSTRIAL USES OF AROMATIC HYDROCARBONS.

The importance of an adequate supply of aromatic hydrocarbons, particularly benzene, toluene, xylene, anthracene, and naphthalene, has been brought prominently to public attention during the past few months through the serious handicap that the European war has placed on American textile manufacturers in the matter of procuring adequate supplies of dyestuffs. Attention also has been directed to the great commercial value of some of these hydrocarbons as basic material in the manufacture of the high explosives now in general demand for military purposes. The industrial importance, however, of these hydrocarbons is by no means limited to the uses mentioned, as the aromatic hydrocarbons are also the source of chemicals important in photography and of valuable medicinal preparations, not to mention creosote oils, solvents, road materials, etc., which are obtained in the course of their refining. Ten crude compounds—benzene, toluene, carbolic acid, anthracene, etc.—which are extracted from the light oils or tars by well-known distillation methods, are raw materials in the manufacture of approximately 300 compounds of extensive commercial use. Figure 1 shows the fractions of average coal tar, and indicates the separations by distillation usually made.

CONSUMPTION AND IMPORTS OF DYESTUFFS.

It is estimated that the value of dyestuffs annually consumed in the United States alone is in the neighborhood of $15,000,000 a year, of which about $3,000,000 worth is supplied by domestic production.[b] Although the difference is perhaps relatively insignificant when compared with figures for other American basic industries, it becomes important when considered in the light of affiliated industries that are largely dependent on these products. It is furthermore an interesting fact that a dozen individual dyes compose well over 75 per cent of the quantity consumed in America.

The great bulk of imported dyestuffs comes from Germany, where the coal-tar chemical industry has attained its greatest development. In the fiscal year 1914 approximately $8,000,000 worth of dyestuffs

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[a] Porter, H. C., Coal-tar products and the possibility of increasing their manufacture in the United States: Tech. Paper 89, Bureau of Mines, 1915, p. 5.

[b] Porter, H. C., Op. cit., p. 6.

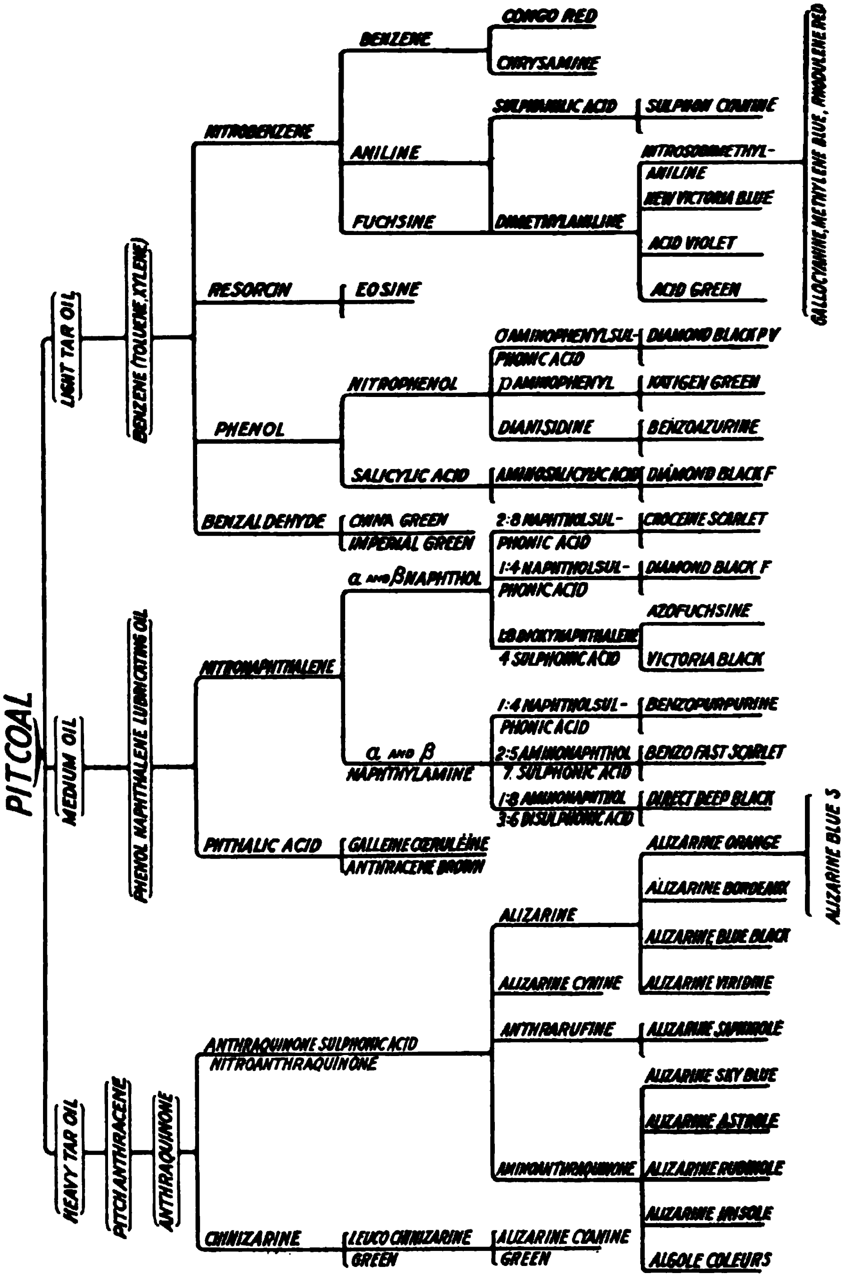

FIGURE 1.—Chart showing some of the products obtainable from the destructive distillation of coal.

was imported from foreign countries,[a] of which Germany supplied nearly $7,000,000 worth. The balance of the imports came from Switzerland, Great Britain, and a few other countries. During the same year, the total consumption of dyestuffs throughout the world amounted to over $90,000,000, of which Germany furnished approximately 75 per cent and in addition furnished over one-half of the material needed to make up the balance.

Germany has controlled not alone the American dyestuff market, but also the dyestuff markets of practically the entire world. Even such countries as England and France, with highly centralized industries and large production of the crude materials necessary for the manufacture of dyestuffs, have been dependent on Germany for their supplies.

CAPACITY OF UNITED STATES TO PRODUCE DYESTUFF BASES FROM COAL.

The potential output of the United States, owing to its vast coal resources and extensive manufacturing interests, is such as to assure ample supplies of the crude materials necessary for the manufacture of dyestuffs, provided an industry be established capable of utilizing the production. During the year 1913, for example, over 69,000,000 tons of coal was coked[b] and approximately 600,000,000 gallons of oil cracked for carbureting water gas. On the basis of average yields this amount of coal and oil was capable of furnishing approximately 550,000,000 gallons of tar and 120,000,000 gallons of light oil.[c] Such quantities of raw material would be more than adequate for the production of greater quantities of coal-tar products than are used in this country.

In the years 1912 and 1913, only about 25 per cent of the total quantity of coke produced was manufactured in by-product ovens.[d] The percentage of by-product ovens to the total number of coke ovens has been rather largely increased since then, and recently a large number of new by-product plants have been built because of the high prices obtained for the recoverable products. The new by-product plants, which were in the process of erection during the early part of 1915, were estimated to increase the output of light oils approximately 14,000,000 gallons a year, so that it is probable that by-product coke ovens are producing to-day nearly 50 per cent of the total coke manufactured in the United States.[e]

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[a] Foreign commerce and navigation of the United States, 1914: U. S Bureau of Foreign and Domestic Commerce, 1915, p. xvii.

[b] Parker, E. W., Manufacture of coke in 1913: Mineral Resources U. S. for 1913, U. S. Geol. Survey, 1914, p. 471.

[c] Porter, H. C., Op. cit., p. 7.

[d] Porter, H. C., Op. cit., p. 7.

[e] Porter, H. C., Unpublished report.

ABNORMAL DEMAND FOR BENZENE AND TOLUENE DURING 1915.

Due to the European war, the demand for benzene and toluene for making explosives has been so great during 1915 as to cause the prices for these hydrocarbons to increase enormously. Toluene, for example, which normally could be bought for less than 25 cents a gallon has netted as high as $8 and $9 a gallon. Benzene, which under ordinary conditions is worth 15 cents a gallon, has brought as high as $1.50 a gallon.

Although the potential production of the aromatic hydrocarbons from coal is sufficient to satisfy the probable demand of the country for years to come, the requisite plants have not yet been built, and necessarily some time must elapse before they can be. By-product coke ovens are expensive and although the profits to be derived from them are considerable, it is probable that the primary cost of construction will make the extension of the number of such plants a matter of progressive growth only. The fact that aromatic hydrocarbons are recovered from coal tars and coke-oven gases purely as by-products makes it improbable that petroleum can compete successfully as a permanent source of supply. The advantage in the use of petroleum lies in the fact that much greater yields can be obtained per unit of original material. Where 1 per cent by weight of light oils can be obtained from coal gas and 0.1 and 0.2 per cent from coal tar on the basis of original coal, petroleum has been proven to yield 12 to 15 per cent and more of the lower boiling aromatic hydrocarbons on the basis of original volume. For example, 100 gallons of oil, worth 3 to 5 cents a gallon, will produce 6 gallons of benzene, 6 gallons of toluene, 2½ gallons of xylene, and smaller quantities of naphthalene and anthracene. Two tons of coal, worth approximately the same amount, will produce 0.7 gallon of toluene, 3 gallons of benzene, and 0.35 gallon of xylene.[a]

The much smaller cost of a plant producing aromatic hydrocarbons from petroleum as compared with the cost of a by-product plant, gives petroleum a considerable advantage as a source of supply for these materials, especially when there is an excessive demand for these products.

The demand for trinitrotoluene by the warring European powers has caused the price of toluene in the United States to soar, although only a small percentage of the trinitrotoluene used has come from this country. It is evident, therefore, that the by-product coke-oven installations and carbureted water-gas plants would to-day be incapable of supplying the toluene that would be needed if our Nation should be called on to go to war. This dearth of supply would continue to exist without such a process as that described herein, as by-product plants could not be constructed quickly enough to meet the Nation's needs. Therefore, the successful large-scale development of such process is a source of national security, as in view of our extensive petroleum resources, the United States could be independent of the world for our supplies of the aromatic bases for the most efficient military explosives.

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[a] Puning, Franz, Benzol, how it is recovered from coal gas: Iron Trade Rev., vol. 53, 1913, pp. 625-632, 653; Wagner, F. H., Coal-gas residuals, 1914, p. 123.

EARLY EFFORTS TO OBTAIN AROMATIC HYDROCARBONS FROM PETROLEUM.

It has long been known that aromatic hydrocarbons could be obtained from petroleum. As early as 1877, according to Veith,[a] Letny[b] obtained a patent for producing aromatic hydrocarbons from petroleum and on that account is regarded as the first experimentor to obtain these products from that source. After Letny, Liebermann and Burg,[c] Salzmann and Wichelhaus,[d] and Lissenko[e] investigated the obtaining of aromatic hydrocarbons from petroleum residues at high temperatures.[f]

The presence of benzene and its homologues in crude petroleum was established at an early date. Prior to 1880 La Rue and Miller had obtained evidence of their presence in a number of crude oils.[g] Benzene has been found in Galician distillates by Pawlewski,[h] Lachowicz,[i] and others; in Baku crude oil by Markownikoff,[j] in crude oil from Tiflis by Beilstein and Kurbatoff; in Rangoon oil by La Rue and Miller, in Roumanian oil by Poui,[k] and in Canadian crude by Schorlemmer. Toluene was also found in the same oils by the same investigators. The proportions of these aromatics in crude oils are, however, small, being only fractions of a per cent. For example, Pawlewski reports having found 0.4 per cent in Galician oil. Manifestly, it would not pay to attempt to extract these small proportions from the oil by distillation methods. Furthermore, investigation has proven the lower aromatic hydrocarbons, benzene and toluene, to be almost entirely absent from American crude oils, although appreciable amounts of aromatic hydrocarbons of higher molecular weight are present, particularly in oils of asphaltic base.

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[a] Veith, Alexander, Gewinnung aromatischer Körper aus den Rückständen: (Bolley and Wiedemann, Handbuch der chemsche Technologie Erdöl) 1862, p. 435; See also Letny, Alexander, Ueber die Einwirkung hoher Temperatur auf Petroleum, Braunkohlentheer und andere ähnliche Stoffe: Dingl. poly. Jour., Bd. 229, 1878, pp. 353-365.

[b] Letny, Alexander, Ueber die Zersetzung des Petroleums und des Braunkohlentheers durch Ueberhitzen: Ber. Deut. chem. Gesell., Jahrg. 11, 1878, pp. 1210–1212; Decomposition of petroleum and lignite tar by superheating: Chem. News, vol. 38, 1878, p. 211.

[c] Liebermann, C., and Burg, O., Ueber die Zersetzung des Braunkohlentheeröls in der Glühhitze: Ber. Deut. chem. Gesell., Jahrg. 11, 1878, pp. 723–726.

[d] Salzmann, M., and Wichelhaus, H., Ueber die Herstellung von Benzol aus Braunkohlentheeröl: Ber. Deut. chem. Gesell., Jahrg. 11, 1878, pp. 802, 1431–1434.

[e] Lissenko, K., Zersetzung des Erdöles beim Erhitzen: Dingl. poly. Jour., Bd. 266, 1887, p. 226.

[f] See Beilstein, F. K., On formation of benzene when petroleum is passed through a hot tube: Handbuch der organ. Chemie, Auflag. 3, Bd. 2, 1896, p. 22.

[g] Petroleum: Encyc. of Chemistry, Lippincott, 1880.

[h] Pawlewski, B., Ueber das Vorkommen des Paraxylols im galizischen Petroleum: Ber. deut. chem. Gesell., Jahrg. 18, 1885, pp. 1915, 1916.

[i] Lachowicz, B., Ueber die Bestandtheile des galizischen Petroleums: Liebig's Annalen, Bd. 220, 1883, pp. 188-206.

[j] Markownikoff, W., and Spady, J., Zur Konstitution der Kohlenwasserstoffe $C_n H_{2n}$ des kaukassischen Petroleums: Ber. Deut. chem. Gesell., Jahrg. 20, Bd. 2, 1887, pp. 1850–1853.

[k] Poui, P., Récherches sur la composition chemique des petroles roumains: Ann. Sci. Univ., t. 2, 1903, pp. 65-80.

The existence of heavier aromatics in a natural state in certain oils, particularly Russian, Roumanian, Californian, and Japanese oils, has led a number of experimenters to attempt to devise methods whereby the yields of hydrocarbons of this series can be greatly increased from such oils. Probably the most work in this field has been done with Russian and Roumanian oils, because of the peculiarly favorable nature of these oils by reason of their content of hydrocarbons of the naphthene type. It is well known to chemists that hexahydrobenzene, for example, will readily split off six hydrogen atoms and leave benzene. This dehydrogenation process is relatively simple and is described in most textbooks of organic chemistry. The character of the reaction involved in making aromatic hydrocarbons from hydrocarbons of the naphthene type, involving a simple splitting off of hydrogen, must not be confused with that involved in making aromatic hydrocarbons from those American petroleums that contain practically no naphthene hydrocarbons.

PROCESSES PATENTED FOR PRODUCTION OF AROMATIC HYDROCARBONS FROM PETROLEUM.

The method devised by Letny [a] involved the passage of petroleum residues obtained from Baku crudes through a highly heated horizontal retort, 7 feet long and 1 inch in diameter, filled with wood charcoal. The residues were allowed to flow through the tubes, which were heated to approximately 700° to 800° C., the vapors coming from the tubes passing to a condenser. The claim was made that 50 to 60 per cent of gas and 40 to 50 per cent of tar was obtained. The tar, on distillation, gave some 17 per cent benzene, 0.4 per cent anthracene, and 7 per cent naphthalene. The oil was fed in slowly, as Letny considered it essential that a comparatively great heating surface be exposed.

Armstrong and Miller [b] reported that their experiments had shown that the liquid formed during the decomposition of petroleum gases contained a mixture of hydrocarbons of the C_nH_{2n-2} series. The liquid was rich in benzene and toluene and showed a trace of the higher aromatic compounds. They advanced the theory that the aromatic hydrocarbons are products in direct line with the paraffins, and are not built up from the acetylene series.

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[a] Letny, Alexander, Ueber die Zersetzung des Petroleums und des Braunkohlentheers durch Ueberhitzen: Ber. Deut. chem. Gesell., Jahrg. 11, 1878, pp. 1210-1212; Decomposition of petroleum and lignite tar by superheating: Chem. News, vol. 38, 1878, p. 211.

[b] Armstrong, H. E., and Miller, A. K., On the decomposition and genesis of hydrocarbons at high temperatures; The products of manufacture of gas from petroleum: Chem. News, vol. 51, June 26, 1885, pp. 307-309.

As previously pointed out, aromatic hydrocarbons have been found to exist in Russian and Roumanian oils; also considerable percentages of naphthene hydrocarbons, which are similar in structure to the benzene hydrocarbons. These naphthenes were contained more largely in the residuum than in the crude oil, and as a result most of the early investigators worked with the residual products.

In 1889 Intschik[a] patented a process for the manufacture of benzene and toluene, in which Russian petroleum residues are dropped into a highly heated vertical chamber where the oil is volatilized and the vapors decomposed. The fixed gases and vapors are drawn off and fractionally condensed.

During the early eighties, when the demand for benzene was high and the supply insufficient to meet the needs of the market, Nobel Brothers, Liebermann and Burg,[b] and Salzmann and Wichelhaus[c] investigated the possibilities of obtaining aromatic hydrocarbons from petroleum residues and are reported to have obtained excellent results.

Liebermann[d] reported in 1888 that the Nobel brothers were erecting a factory in the Baku field for the purpose of manufacturing aromatic hydrocarbons from petroleum residues.

Hlawaty,[e] in 1888, patented a process by means of which he claimed to obtain rich mixtures of benzene and its homologues, through the action of highly heated steam, or ethylene, methylethylene, or alcohol on petroleum residues, the mixed vapors being passed through red-hot tubes filled with charcoal and wood turnings.

A patent with the same object in view, granted to Dvorkovitz[f] in 1892 proposes to decompose steam passed into a gasifier in which the liquid hydrocarbon is vaporized. The water gas is there carbureted and passed into a condensing chamber where the benzene, toluene, and other aromatic compounds are separated.

A later patent issued to Dvorkovitz[g] states that the liquid hydrocarbon flows through a series of retorts in which the liquid is progressively subjected to temperatures ranging from 300° to 1,000° C. The retorts, which are wide but shallow, are inversely inclined so that the lower end of each retort communicates with the upper end of the retort below it. The vapors from the lower retort are then passed to a condenser.

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[a] Intschik, —, Russian Patent issued in 1889.

[b] Liebermann, C., and Burg, O., Ueber die Zersetzung des Braunkohlentheeröls in der Glühhitze: Ber. Deut. chem. Gesell., Jahrg. 11, 1878, pp. 723–726.

[c] Salzmann, M., and Wichelhaus, H., Ueber die Herstellung von Benzol aus Braunkohlentheeröl: Ber. Deut. chem. Gesell., Jahrg. 11, 1878, pp. 802, 1431–1434.

[d] Liebermann, C., Neue Gewinnungsmethode von Benzol, Naphtalin, und Anthracen: Dingl. Poly. Jour., Bd. 246, 1882, pp. 429–432.

[e] Hlawaty, Faustin, German Patent 51553, Aug. 11, 1888.

[f] Dvorkovitz, Paul, English Patent 21647, Nov. 26, 1892; also, Dvorkovitz, Paul, The distillation, cracking, and gasification of petroleum hydrocarbons: Petroleum Rev., vol. 17, Sept. 28, 1907, pp. 176–179; Gasification of coal and liquid hydrocarbons: Jour. Soc. Chem. Ind., vol. 12, May 31, 1893, pp. 403–412.

[g] Dvorkovitz, Paul, English Patent 23671, Dec. 5, 1894.

Nikiforoff,[a] who has long been one of the most noted and brilliant workers in this field, has patented a process for benzene-toluene manufacture involving two-stage decomposition under varying conditions. The crude oil is fed into a series of decomposition retorts, the course of the liquid being obstructed by baffle plates so as to prolong the action of the heat. A temperature of 500° to 525° C. is maintained without pressure. The vapors pass through these retorts to a separating tank maintained at a temperature of 200° to 250° C., where the heavier products are condensed. The more volatile products (products boiling at less than 200° C.), which remain uncondensed at that temperature, are then conducted to a separate retort where they are subjected to a temperature of 900° to 1,000° C. and to a pressure of about 2 atmospheres (30 pounds per square inch).

In a later patent Nikiforoff[b] proposes to obtain aromatic hydrocarbons by the coking of naphtha in a metallic retort provided with troughs along which the naphtha flows, the hydrocarbon vapors given off below 200° C. to be used in carbureting water gas. Air is blown through heated coke or anthracite to obtain a generator gas. Superheated steam is blown into the generator gas to obtain a water gas. The water gas is then carbureted with the decomposition products of the naphtha. The heated water gas and hydrocarbon vapors are placed under a pressure of not less than 1 atmosphere (15 pounds per square inch.) The gas and vapors are passed through a washing apparatus, where the water and heavier hydrocarbons are collected, and the gas and vaporous hydrocarbons boiling under 200° C. pass on to a condenser, which separates the liquid aromatic products and allows the gas to pass on to a collector.

In 1897, Pamfilow[c] was granted a patent[d] for a process involving the passage of petroleum residues through a preheater into a vaporizer, and then into a coil of many turns and great length, where the vapors are exposed to gradually increasing temperatures. He claims the production of a tar, consisting almost exclusively of aromatic hydrocarbons, which upon distillation gives 15 per cent benzene, 1 per cent naphthalene, and 0.5 per cent anthracene on the basis of the original oil.

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[a] Nikiforoff, Alexander, Russian Patent 290, Dec. 31, 1894; German Patent 85, Mar. 12, 1895; French Patent 315428, Oct. 29, 1901; English Patent 10957, Aug. 27, 1886; U. S. Patent 755309, Mar. 22, 1904.

[b] Nikiforoff, Alexander, English Patent 17450, July 30, 1907.

[c] Pamfilow, S. M., Gewinnung von Leuchtgas und aromatischen Kohlenwasserstoffen aus Erdöl: Chem. Ztg., Jahrg. 21, Bd. 2, 1897, p. 1069.

[d] Russian Patent 322, Sept. 13, 1897.

Zelinsky[a] and Ogloblin[b] report the obtaining of excellent results by Nikiforoff's process. The former, however, employed the method only on a small scale, treating several liters of petroleum a day. They report having been able to obtain a fraction boiling between 75° and 180° C., which represented 14.1 per cent of the original oil, and contained 58 per cent of benzene and 28 per cent of toluene.

Hausman and Pilat[c] proposed a process for producing aromatic hydrocarbons that involves the introduction of "naphtha hydrocarbons" in a vaporized form into heated tubes placed at a slight inclination in a long combustion furnace, such tubes being filled with any suitable catalytically active material, such as oxides of lead, manganese, etc. Air or superheated steam is forced into the tubes by means of a pump, so that the free hydrogen from the hydrocarbons and the oxygen from the air or steam may unite.

Holcgreber,[d] in a late patent, proposes to pass petroleum in the form of vapor, together with an excess of pure hydrogen, through pipes filled with suitable catalytic agents, such as iron, copper, nickel, or cobalt. The catalytic agents are used alone or mixed with an inert material such as asbestos. The tubes are kept at a temperature of 180° to 300° C.

Another recent patent along somewhat different lines is that granted to Starke[e] for the production of benzene and its homologues from petroleums, particularly Japanese and California petroleums. The petroleum distillate containing the benzene and its homologues is mixed with concentrated sulphuric acid, which is heated and agitated until the evolution of sulphur dioxide has ceased. The mass is then allowed to settle until the sulphonic acids have separated from the petroleum, and the benzene is then recovered by ordinary distillation.

Other processes of the same type as that last mentioned are those patented by the Disconto Gesellschaft[f] in Berlin and by Tammann.[g]

Testelin and Renard[h] and Meffert[i] propose to use a mixture of superheated steam and oil, which is then highly heated, to produce aromatic hydrocarbons. Testelin and Renard pass the mixture over a layer of red-hot clay, whereas Meffert heats the mixture in refractory retorts to a temperature of 1,200° to 1,400° C. Neither process employs pressure. In fact Testelin and Renard claim that the use of pressure is prejudicial.

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[a] Zelinsky, N. D., Vortrag über die Darstellung aromatischer Kohlenwasserstoffe aus dem Erdöl (Preparation of aromatic hydrocarbons from petroleum): Chem. Ztg., Jahrg. 26, Bd. 1, 1902, p. 68; Jour. Soc. Chem. Ind., vol. 21, Feb 15, 1902, p. 166.

[b] Ogloblin, W. N., Die Darstellung von Benzol und seiner Homologen aus russischem Naphta nach Verfahren von A. N. Nikiforoff: Chem. Central-Blatt., Jahrg., 75 Bd. 2, 1904, p. 830.

[c] Hausman, Joachim, and Pilat, Stanislaus, German Patent 227178, April 1, 1909.

[d] Holcgreber, Jean, English Patent 17272, July 28, 1913.

[e] Starke, E. A, U. S. Patent 913780, Mar. 2, 1909. See also U. S. Patent 1109187, Sept. 1, 1914.

[f] Disconto-Gesellschaft in Berlin, German Patent 216459, May 23, 1908.

[g] Tammann, Gustav, German Patent 95579, Mar. 3, 1897.

[h] Testelin, Auguste, and Renard, Georges, German Patent 268176, Aug. 11, 1908.

[i] Meffert, Friedrich, German Patent 99254, Mar. 13, 1897.

DISTINCTION BETWEEN PRIOR PROCESSES AND THOSE DESCRIBED HEREIN.

References to a number of processes for benzene-toluene production not herein reviewed are contained in the bibliography at the end of this report. Those processes particularly mentioned here indicate sufficiently the prior development of the art, and the lines along which the problem has been approached.

The processes reviewed are primarily adapted to and dependent on the use of oils that are of a highly favorable character, and in connection with which the reaction necessary for benzene-toluene production is comparatively simple. In other words, the oils used were of the special varieties most favorable for the production of aromatic hydrocarbons. Either they contained the polycyclic (asphaltic) compounds that decompose on simple splitting into benzene and its homologues, or else naphthenes were present, which yield aromatic hydrocarbons through the simplest sort of dehydrogenation. Practically all of these prior processes are operated at atmospheric pressure, indicating the simplicity of reaction involved, as compared with the production of the low-boiling aromatic compounds from oils in which naphthenes and aromatics do not naturally occur, as accomplished by the process described later. The difference is that between a relatively simple alteration and an extensive and complex rearrangement.

In none of the descriptions of these prior processes is there the slightest mention of their availability for transforming hydrocarbons of the paraffin type into aromatic hydrocarbons, which is one of the characteristic advantages of the process discussed later. Otherwise stated, none of these processes is suitable for all types of oil. Although, as has been previously pointed out, it has long been known that benzene may be formed by the passing of petroleum at atmospheric pressure through a red-hot tube,[a] the quantities of benzene thus obtained are relatively small as compared with the quantities of by-products of little or no value, so that such processes, so far as is known, have not succeeded in industrial operations. This fact is pointed out by Hausman and Pilat,[b] and others.

THEORIES OF FORMATION OF AROMATIC HYDROCARBONS.

In regard to the various theories concerning the character of reaction involved in producing aromatic hydrocarbons from petroleum hydrocarbons, as exemplified in processes of the type reviewed, there seems to be considerable difference of opinion. The three views most commonly discussed, which are probably all measurably correct, depending on the type of oil used and the conditions of time, temperature, and pressure in the reacting zone, are:

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[a] Beilstein, F. K., Handbuch der organischer Chemie, Auflage 3, Bd. 2, p. 22.

[b] Hausman, Joachim, and Pilat, Stanislaus, German Patent 227178, Apr. 1, 1909.

(1) That there is a splitting off of cyclic nuclei;[a]

(2) That polymerization of ethylene and acetylene hydrocarbons occurs;[b]

(3) That dehydrogenation of naphthenes occurs.[c]

OBJECT AND SCOPE OF BUREAU'S EXPERIMENTS WITH CRACKING PROCESSES.

The experimental work that resulted in the discovery of the processes described in this report was undertaken for the purpose of studying the reactions that occur in the cracking of liquid hydrocarbon mixtures.

The object of the bureau's experimental work, broadly stated, was to determine the effect of various combinations of temperature and pressure on the course of the cracking reaction, and to determine, if possible, *the most favorable conditions for the maximum yields of the various desired cracked products*, such as gas, gasoline, and low-boiling aromatic hydrocarbons.

The first phase of the work dealt with the production of ethylene hydrocarbons used in carbureting water gas.[d] In the production of carbureted water gas, petroleum distillates are commonly employed to increase the illuminating power of the product, and the increasing cost of "gas oils" has been a serious handicap to the continued existence of the carbureted water-gas industry.

Four to five gallons of oil is required per 1,000 cubic feet of carbureted gas produced. Consequently the addition of 1 cent per gallon at once becomes a serious matter to gas works, particularly in view of the fact that the numerous cracking processes for the production of gasoline will require the use of gas oil as the basic material, thereby greatly broadening the market for the latter, with resulting higher cost. Furthermore asphaltic distillates are not particularly desirable for use in carbureting gases. In fact the problem has become so serious that many gas men strongly favor coal-gas processes despite the fact that 75 per cent of the artificial gas produced in America to-day is carbureted water gas.

In the course of the experiments it was found that highly favorable conditions were obtained by having relatively high temperatures and low pressure in the cracking zone. The influence of pressure is clearly illustrated by the data in Table 1 following and by the curves shown in figures 2 to 5.

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[a] Brooks, B. T., Bacon, R. F., and others, The preparation of gasoline and kerosene from heavier hydrocarbons: Jour. Ind. and Eng. Chem., vol. 7, Mar., 1915, p. 180; Lewes, V. B., The production of oil-gas from Russian petroleum: Jour. Soc. Chem. Ind., vol. 11, July 30, 1892, pp. 584–590.

[b] Ipatiew, W., Polymerisation der Äthylen-Kohlenwasserstoffe bei hohen Temperaturen und Drucken: Ber. Deut. chem. Gesell., Jahrg. 44, Bd. 3, 1911, pp. 2978–2987; Meyer, Richard, Pyrogene Acetylen-Kondensationen: Ber. Deut. chem. Gesell., Jahrg. 45, Bd. 2, 1912, pp. 1609–1633.

[c] Beilstein, F. K., Loc. cit.; Lewes, V. B., Loc. cit.

[d] Whitaker, M. C., and Rittman, W. F., Thermal reactions in carbureting water gas: Jour. Ind. and Eng. Chem., vol. 6, 1914, pp. 383–392, 472–479; Jour. Soc. Chem. Ind., vol. 33, July 15, 1914, p. 682.

Table 1.—*Results of cracking oil under pressures of 3 atmospheres to about one-thirtieth of an atmosphere, showing effect of various pressures on gas formation.*

Temperature.	Oil used.	Absolute pressure.	Carbon.	Tar.	Gas.	Illuminants.	Methane. (CH_4).	Ethane (C_2H_6).	Hydrogen. (H_2).
°C.	*C. c.*	*Pounds.*	*Grams.*	*C. c.*	*Liters.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
650	400	45	8	133	145	30.5	45.1	11.5	9.3
750	400	45	26	87	194	15.5	56.6	6.1	17.5
900	400	45	165	9	310	5.0	41.6	None.	50.0
		Atmosphere.							
650	400	1	3	163	135	43.6	33.7	10.2	9.1
750	400	1	18	80	206	30.6	41.1	4.9	19.2
900	400	1	115	11	382	13.1	46.7	None.	38.6
750	400	1/20–1/30	1	153	146	56.1	....	....	12.5
850	400	1/20–1/30	3	100	211	52.9	20.5	3.4	15.6
900	400	1/20–1/30	3	60	234	52.1	24.0	1.3	17.3
950	400	1/20–1/30	12	58	235	46.9	27.0	Trace.	20.8

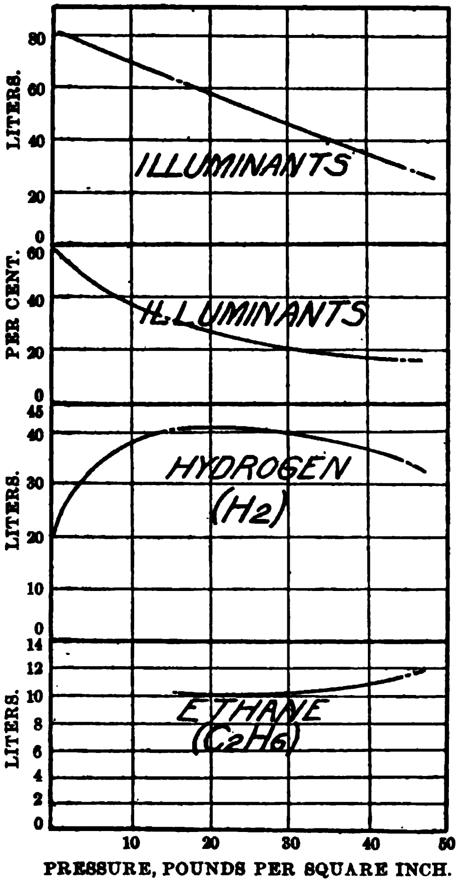

Figure 2.—Curves showing effects of pressure on proportions of illuminants, H_2, and C_2H_6 from oils cracked at 750° C.

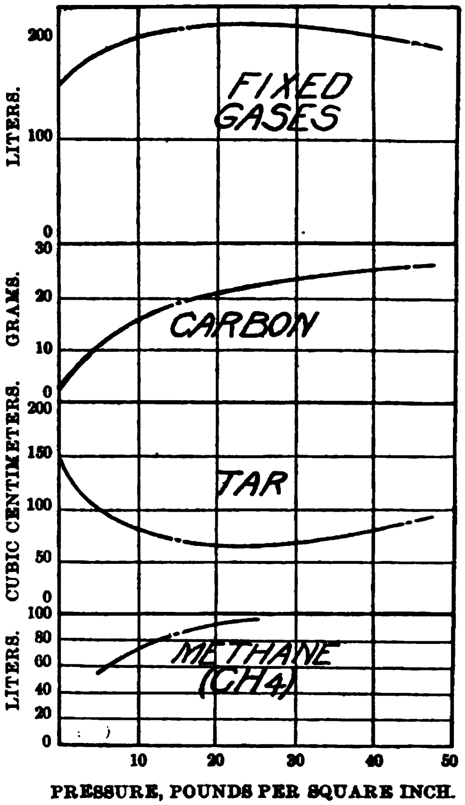

Figure 3.—Curves showing effects of pressure on proportions of fixed gases, carbon, tar, and CH_4 from oils cracked at 750° C.

In this research qualitative data were obtained showing the marked influence of the presence of different end-product gases on the cracking of oil. The results are shown in Table 2 following. The actual diminution of added hydrogen should be noted.

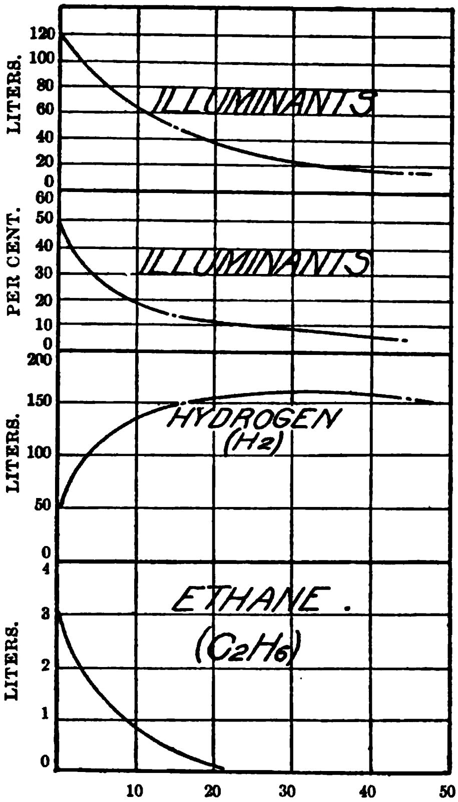

FIGURE 4.—Curves showing effects of pressure on proportions of illuminants, H_2, and C_2H_6 from oils cracked at a temperature of 900° C.

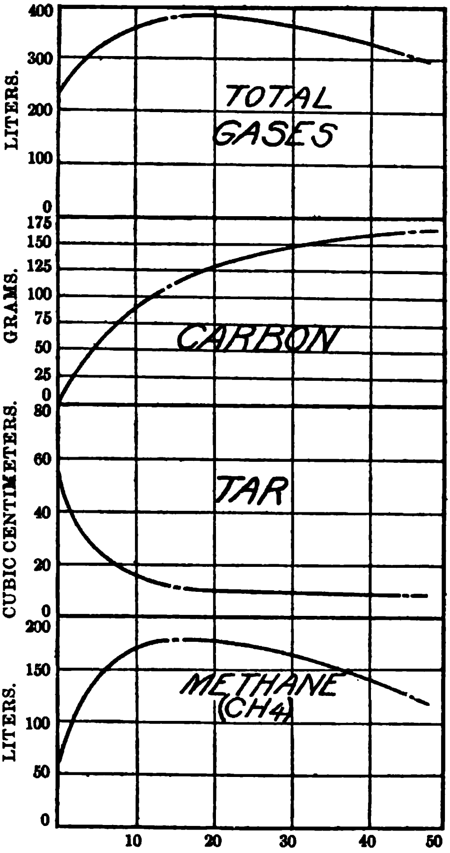

FIGURE 5.—Curves showing effects of pressure on proportions of fixed gases, carbon, tar, and CH_4, from oils cracked at 900° C.

TABLE 2.—*Results of cracking oil in an atmosphere of hydrogen, showing effect on gas formation (compare with Table 1).*

Temperature.	Absolute pressure.	Quantity of oil used.	Admixed hydrogen.	Ethane (C_2H_6).	Methane (CH_4).	Illuminants.	Residual hydrogen (H_2).	Shrinkage in hydrogen.
° C.	*Pounds.*	*C. c.*	*Liters.*	*Liters.*	*Liters.*	*Per cent.*	*Liters.*	*Liters.*
750	15.0	400	358	15.4	125.0	70.6	308	50
800	15.0	400	412	18.0	116.0	83.2	335	77
750	0.75	400	400	9.5	52.0	112.0	381	19
810	0.75	400	413	....	86.5	140.0	378	35
860	0.75	400	388	....	99.5	133.0	350	38
900	0.75	400	292	....	92.0	120.0	272	20
960	0.75	400	382	....	95.0	113.0	348	34

A summation of the results obtained in this first stage of the investigation of the general problem is shown in Table 3 and in figures 6 and 7

TABLE 3.—*Summary of results of oil-cracking experiments, showing effect of varying temperatures and pressures and of admixtures of hydrogen on gas formation.* (*See Tables 1 and 2.*)

[All results based on 400 c. c. of oil and calculated to 0° C. and 760 mm. pressure.]

ATMOSPHERIC-PRESSURE GROUP.

Furnace conditions.		Reaction products.							Hydrogen admixed.	Hydrogen shrinkage.
Temperature.	Pressure per square inch.	Gas.	Carbon.	Tar.	Illuminants.	Methane (CH_4).	Ethane (C_2H_6).	Hydrogen (H_2).		
°*C.*	*Pounds.*	*Liters.*	*Grams.*	*C. c.*	*Liters.*	*Liters.*	*Liters.*	*Liters.*	*Liters.*	*Liters.*
650	15.0	135	3	163	58.8	45.5	13.8	12.1	....	....
750	15.0	206	18	80	63.0	84.5	10.15	39.6	....	....
900	15.0	382	115	11	50.0	178.1	Trace.	148.2	....	....
HIGH-PRESSURE GROUP.										
650	45.0	145	8	133	44.3	65.2	16.7	13.1	....	....
750	45.0	194	26	87	30.1	110.0	11.8	33.9	....	....
900	45.0	310	165	9	15.5	128.9	None.	155.0	....	....
LOW-PRESSURE GROUP.										
750	0.75	146	1	153	82.0	....	....	18.3	....	....
850	.75	211	8	100	115.5	43.2	7.16	32.9	....	....
900	.75	234	3	60	122.0	56.0	3.00	40.0	....	....
950	.75	235	12	58	110.0	63.4	Trace.	48.8	....	....
ADMIXED-GAS GROUP.										
750	15.0	....	....	....	70.6	125.0	15.4	308.0	358	50
800	15.0	....	....	....	83.2	116.0	18.0	335.0	412	77
750	1.0	....	....	....	112.0	52.0	9.5	381.0	400	19
810	1.0	....	....	....	140.0	86.5	....	378.0	413	35
850	1.0	....	....	....	133.0	99.5	....	350.0	388	38
900	1.0	....	....	....	120.0	92.0	....	272.0	292	20
950	1.0	....	....	....	113.0	95.0	....	348.0	382	34

This work, dealing with the field of oil-gas production, constituted a first step of the study of the cracking reactions for the production of gas, gasoline, aromatic hydrocarbons, and other products. Acknowledgment is due to Prof. Milton C. Whitaker, of Columbia University, for his active cooperation and assistance in the study of the broad problem. The original plan was that Professor Whitaker and one of the authors should continue joint work in this field, but the employment of the latter by the Bureau of Mines rendered the fulfillment of this plan impossible. To Prof. Whitaker, however, credit should be given for his active interest in the present researches and his recognition of the commercial importance of the results obtained.

MANNER OF APPROACHING SOLUTION OF CRACKING PROBLEM IN LABORATORY EXPERIMENT.

The nature of the problem has already been indicated. It was at once foreseen, however, that mechanical difficulties would be encountered if experiments were attempted along conventional lines. The disadvantages of all methods involving the heating of liquid and vapor simultaneously are evident. The interdependence of temperature and pressure limits the conditions under which reactions may be studied, and in addition attempts to heat considerable quantities of liquid involve a large element of danger. It soon appeared, however, that all these disadvantages could be obviated by eliminating the liquid phase from the cracking system, and that no new difficulties would be introduced by the elimination. The method of procedure developed is absolutely flexible with regard to the variation of experimental conditions and permits work with a

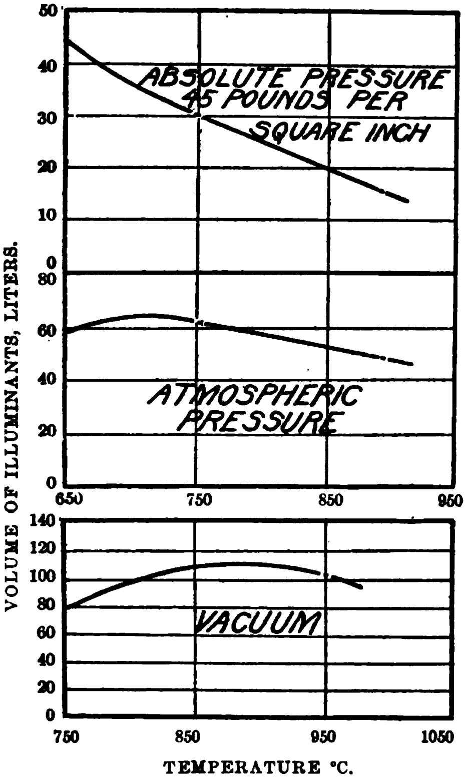

FIGURE 6.—Curves showing effects of temperature and pressure on volume of illuminants in oil-gas cracking process.

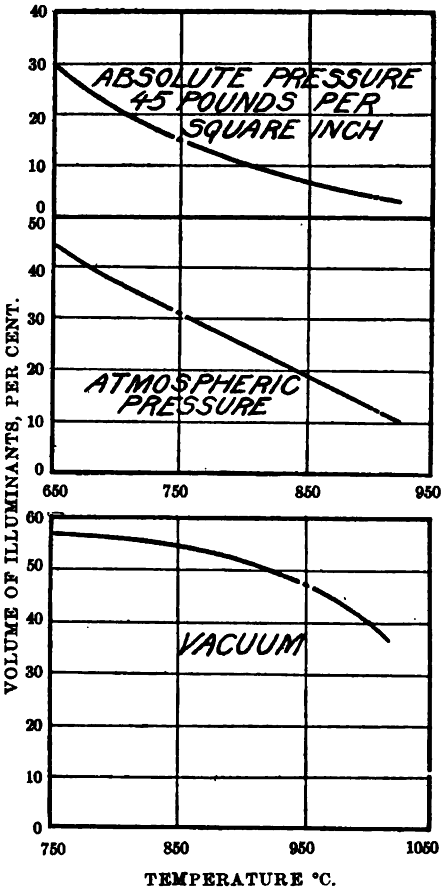

FIGURE 7.—Curves showing effects of temperature and pressure on proportions of illuminants in oil-gas cracking process.

minimum of danger. The actual nature of the reaction was given less consideration than the commercially important questions of initial materials and final products and the regulation of conditions necessary to produce desired ends.

NECESSITY FOR COOPERATION OF CHEMISTS, ENGINEERS, AND PRACTICAL REFINERS.

There are few industrial operations coming under the field of physical chemistry that are more complex in character than that involved in the treatment by heat of hydrocarbons for transformation into various products. Practically all the variables of chemical phenomena are involved, and the complexity of the reactions is such that little, if anything, is known about a great many of them. Although the problem is thus complicated, both as regards theory and practice, the possibilities are great.

Chemical development has lagged behind both mechanical and operative improvements in the petroleum industry. The explanation can be found in the fact that the solution of too many problems of theoretical chemistry has been left to the mechanical engineer or practical operator, who can not be expected to be as efficient in chemical research as a man who has devoted himself to the study of chemical and physical phenomena and their industrial applications.

Only the combination of the skill of the mechanical engineer with the detailed knowledge of the chemist can yield the best results. It is obvious that maximum efficiency can no more be obtained by a mechanically perfect machine designed on wrong principles than by one that is mechanically weak though theoretically correct.

PLACE AT WHICH BUREAU'S EXPERIMENTS WERE CONDUCTED.

The experimental work described in Part I of this report was done in the department of chemical engineering of Columbia University, New York, which extended the facilities of the laboratories, libraries, and other equipment for use in experiments.

CHARACTER OF EXPERIMENTS.

The work consisted essentially in the subjection of five typical petroleum oils to a wide range of conditions of temperature and pressure. The oils used were as follows:

1. A water-white Pennsylvania kerosene or burning-oil distillate, representing the paraffin-base oils of the Appalachian oil fields.
2. An Oklahoma residuum, typifying the mixed-base oils of the Mid-Continent field.
3. A California (Kern River) crude, representing the asphaltic-base oils of the Pacific Coast fields.

4. A Mexican crude.
5. A Russian residuum.

The general course of procedure in the experiments was as follows:

The oil was subjected to treatment at various temperatures ranging from the initial boiling point of the material to as high as 1,000° C., the increase being in stages of 150° C. In like manner the effect of pressure was studied over a range varying from about one-thirtieth of an atmosphere to a pressure higher than 30 atmospheres (500 pounds per square inch). Various combinations of temperature and pressure were applied systematically to each oil.

It is to be understood that work was carried on with the idea of obtaining comprehensive results rather than for the purpose of discovering optimum conditions for the production of any desired hydrocarbons. Unfavorable and favorable conditions were studied with equal care. Attention is especially called to this feature, because many unfavorable data are necessarily recorded, and these might be considered as militating against the commercial practicability of the results.

GENERAL DISCUSSION OF GASOLINE AND BENZENE-TOLUENE PROCESSES.

The processes more particularly described herein have been commonly termed a gasoline process and a benzene-toluene process. In both the general method of operation is similar, but the conditions of control differ materially and the end products vary widely. The gasoline process is operated at lower temperatures and high pressures; for example, at a temperature of 500° to 550° C. and at a pressure up to 300 pounds, whereas the benzene-toluene process is operated at a temperature of 625° to 675° C. and at a pressure up to 250 pounds. However, the reader will understand that if the rate of oil feed is properly varied, it is not necessary to work within the ranges of temperature and pressure specified.

The processes are to be distinguished from the ordinary cracking processes in that the hydrocarbon material is kept in a gaseous state throughout the entire course of operation. In processes involving the subjection of large masses of liquid to high temperatures and the pressures necessary to prevent volatilization, degrees of heat exceeding 400° to 450° C. are dangerous because the corresponding pressures are excessive.

ADVANTAGES OF GASEOUS-PHASE CRACKING PROCESSES.

The advantages of having the reaction occur entirely in the gaseous state are fourfold, as follows:

First. The hydrocarbon material is fully expanded, so that if there is a leakage of joints or a giving way of the retainer no danger results.

Second. Much higher temperatures and pressures can be used than in liquid or liquid-vapor processes.

Third. The speed of reaction incident to high temperatures can be readily attained.

Fourth. Control conditions can be varied at will.

CHEMICAL FACTORS IN GASEOUS REACTIONS.

The chemical factors that influence equilibrium in any gaseous chemical reaction are as follows:

1. Temperature.
2. Pressure.
3. Concentration (mass action).
4. Duration.
5. Contact surface (catalysis).

EQUILIBRIUM.

True equilibrium is considered to be that state in which reactions proceed equally in each direction. Under constant pressure there is a definite proportion of the individual constituents for each temperature. Likewise for each pressure, under constant temperature, there is a definite proportion between the reacting hydrocarbons. This in spite of the fact that the numerical value of the equilibrium constant is a function only of temperature. Although the action of heat can impart the energy necessary to disturb an existing state of chemical inertia and induce reaction between the various molecular formations, too high temperatures for a period of time will defeat the end sought by permitting the hydrocarbon to decompose into coke and permanent gases.

It is well known that the tendency of substances to react depends on the nature of the substances and the distance of the system from stable equilibrium. This tendency is offset by chemical inertia, which decreases with increasing temperature. Also to be considered is the fact that increasing the temperature may bring the system farther from or nearer to the desired equilibrium point.

BEARING OF THEORETICAL EQUILIBRIUM RELATIONS ON INDUSTRIAL OPERATIONS.

Equilibrium values, therefore, are of high importance in industrial operations as they indicate the direction and magnitude of reactions. Knowledge that a given reaction will be attended by evolution, or absorption of heat, is of aid in determining operating conditions. No single equilibrium, however, can be considered by itself, as not only must a particular equilibrium relationship be satisfied, but all the constituents must also be in equilibrium. In other words, there must be a symphony of equilibria between all the components of the system. The moment there is an unbalanced system of gases there is a tendency for an equilibrium to be established between all the components of that system. All the reactions that occur in a hydrocarbon process are interrelated and must all be taken into consideration, although a single reaction or set of reactions may be extremely important as indicating a prevailing tendency.

RESULTS OF CRACKING IN AN ATMOSPHERE OF ADMIXED GASES.

If petroleum is cracked in an atmosphere containing all the hydrocarbon gases with the exception of ethylene, one may expect that the only fixed gas produced in any quantity will be ethylene, at least until there is ethylene enough to satisfy equilibrium conditions between all the constituents present in the reaction chamber at the given temperature of operation. Advantage is taken of this fact in the processes described herein, as the fixed gases are retained in the reaction zone so that equilibrium conditions with respect to these gases are continuously satisfied without further decomposition for their formation.

In general, in the operation of the processes two classes of gases are formed—first, those that are readily condensable at temperatures above, say, 30° C., and second, those that remain uncondensed below 30° C., such as hydrogen, ethane, methane, and like hydrocarbon gases, which are herein termed fixed or permanent gases. If the condensable gases, so called, be continually withdrawn, and if oil, which is gasified, be constantly supplied, and if the permanent gases, or a considerable part thereof, be held in the sphere of reaction, there is a tendency for the equilibrium to be restored and for condensable hydrocarbons rather than noncondensable or permanent gases to be formed.

Thus it is not necessary to generate new gaseous end products at the expense of more valuable condensable hydrocarbons. The continuous removal of the condensable products from the cracking zone aids the establishing of equilibrium by the continual formation of these products. What is attempted is to have the reaction products approach a state of equilibrium with a constant removal of the liquid end products, so that the reaction will proceed continuously in the desired direction. Stated in another way, the addition of any of the end products in any decomposition process checks decomposition in that direction and causes it to proceed in another direction until equilibrium has been established. Continuous removal of the products of reaction from the reaction chamber is merely in obedience to the indications of the law of mass action and permits the reaction to proceed to approximate completion.

EFFECT ON CRACKING REACTION OF HIGH CONCENTRATION OF END PRODUCTS.

The tendency mentioned above is promoted by a high concentration or increase in the density of the permanent gases that are retained in the sphere of reaction. Concentration of a given gas is a function of pressure, but the character of the concentration products exerts a material influence on the reaction products.

The relation between pressure and concentration can best be understood from the well-known thermodynamic relationship for any temperature in which—

Pressure × volume = constant
volume = 1 divided by concentration.

The thermodynamic relationship can be expressed by the equation

Pressure = concentration × constant,

from which it becomes evident that the concentration of end-product hydrogen and other permanent gases is directly proportional to the pressure exerted by such gases.

In the ordinary cracking processes some decomposition of the hydrocarbon material is necessary to produce hydrogen and other gases required to establish an equilibrium at the temperature of operation. The degree of this extreme decomposition might, of course, be limited by the addition of the necessary hydrogen and the other gases from extraneous sources. However, as free hydrogen is costly, such a procedure is commercially impracticable. Some investigators have sought to attain the same end by the decomposition of steam.

In the experiments resulting in the discovery of the process for the manufacture of gasoline, it was found that if oil is cracked in the presence of an excess of the hydrogen and other permanent gases produced by the process itself and retained in the zone of reaction, little of the hydrogen contained in the oil splits off in a free condition. In fact, when ethylene is sought, a considerable part of the added hydrogen enters into combination and produces more hydrocarbons than would otherwise be obtainable. The value of this feature lies in the fact that there is no necessity of generating new gaseous products at the expense of the more valuable substances. With more hydrogen present than is required by equilibrium conditions the natural tendency is for the hydrogen to combine. This feature constitutes one of the essentials of both processes. The general effect of changed concentration of one of the constituents in an equilbrium has been discussed by one of the authors.[a] The important bearing of the atmosphere in which the decomposition of oil occurs has been noted by other experimenters. Hempel,[a] for example, found that when oil is cracked in an atmosphere of hydrogen none of the hydrogen is split off from the hydrocarbons, and a part of the admixed hydrogen actually combines for the formation and preservation of hydrocarbons. The result of experiments conducted by one of the authors[b] fully confirms this; furthermore the same principles have been successfully applied in gas manufacturing by the Joneses on the Pacific coast. Hempel also found that the greater part of the hydrogen admixed enters into combination to form saturated hydrocarbons when cracking is done under pressure. This fact likewise has been confirmed by one of the authors.

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[a] Rittman, W. F., Application of physical chemistry to industrial processes: Jour. Ind. and Eng Chem., vol. 6, August, 1914, p. 684.

TEMPERATURE.

As organic substances generally decompose on application of heat, it is obvious that increase in temperature favors a high degree of decomposition. Heavy molecules are less stable than light ones of similar structure. For example, methane is stable at 400° C., whereas at that temperature a higher member of the same series, such as paraffin wax, undergoes considerable decomposition. Thus the selection of temperature in the processes under discussion depends on the character of the hydrocarbon materials to be treated, as decomposition of each group of hydrocarbons is most favored by a specific temperature. In this respect these processes have a material advantage, because there are no mechanical limitations regarding the character of hydrocarbons that may be employed as original material. Any petroleum product, from gasoline to the heaviest residuum, can be utilized for the production of low-boiling aromatic hydrocarbons or gasoline. Of course, in the production of gasoline, higher-boiling hydrocarbons than those commercially known as gasoline would be utilized, as there would be no object in cracking a natural gasoline, although such cracking would be possible.

RELATION OF TEMPERATURE TO PRESSURE.

Indications of the effect of temperature on cracking processes may be obtained from thermodynamics. According to a proposition put forward by Bertholet and Thomsen, "every chemical change gives rise to those substances that occasion the greatest development of heat." If this were true, the problem of obtaining temperature indications would be as simple as that of obtaining pressure indications for reactions proceeding to equilibrium.

To-day it is recognized that this statement is only a first approximation, because in all reactions one deals with the additional molecular energy, whereas the proposition mentioned assumes the free energy, termed maximum work, to be equal to the total energy change. Nernst has pointed out that this statement holds true only at the absolute zero; that is, the entropy of liquids and solids at absolute zero of temperature equals zero. The practical significance of a relationship such as that indicated by the Van't Hoff equation showing the relative values of equilibrium constants for two different temperatures, and the Nernst formula giving an approximate absolute value of the equilibrium constant for a given temperature are fully discussed by Whitaker and Rittman.[a]

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[a] Hempel, H., Über Gasöle und Ölgas: Jour. Gasbel, Jahrg. 53, Jan. 15, 1910, pp. 53-58.

[b] Whitaker, M. C., and Rittman, W. F., Thermal reactions in carbureting water gas: Jour. Ind. and Eng. Chem., vol. 6, 1914, pp. 383-392; Jour. Soc. Chem. Ind., vol. 33, July 15, 1914, p. 682.

It is fully recognized that constants obtained by the Nernst formula are not final, and are of qualitative value only. It is likewise recognized that practically all of the equilibrium constants indicated and used in the earlier work cited above are at best approximations, but the quantitative numerical results derived serve as indicators and not as specific and invariable quantitative factors. If these facts be recognized at all times, and if it be further remembered that every individual reaction is influenced by every other reaction possible in the zone of reaction, most helpful information may be derived. Without such constants the effects of temperature and pressure are unknown factors, to be determined largely in an empirical way. The theoretical considerations assist in arriving at an intelligent understanding of the results obtained, together with further suggestions as to the most likely next step. As illustration, a number of equilibrium relations used in the course of the various researches covered by this report are listed in Table 4, following:

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[a] Whitaker, M. C., and Rittman, W. F., Thermal reactions in carbureting water gas: Jour. Ind. and Eng. Chem., vol. 6, May and July, 1914, pp. 383, 472.

TABLE 4.—*Results of quantitative study of equilibria.*[a]

[If no heats of reaction could be found in the literature, as in the case of the higher aliphatic hydrocarbons, these values have been calculated according to the empirical formulas based on the theory of linkage as outlined by Thomsen, J., Thermochemischer Untersuchungen, pt. 4, chap. 14.]

GAS REACTIONS.

Reaction.	Heat of reaction.	Volume change.	Equilibrium constant.	Value of constant.		
				K_{600}	K_{750}	K_{900}
$C + 2H_2 \rightleftarrows CH_4$ Carbon Hydrogen Methane.	+18900	2 to 1	$K=\frac{CH_4}{(H_2)^2}$	0.077	0.012	0.003
$2C + 3H_2 \rightleftarrows C_2H_6$ Carbon Hydrogen Ethane	+23300	3 to 1	$K=\frac{C_2H_6}{(H_2)^3}$	2.2×10^{-7}	1.7×10^{-8}	2.5×10^{-9}
$2C + 2H_2 \rightleftarrows C_2H_4$ Carbon Hydrogen Ethylene	−14600	2 to 1	$K=\frac{C_2H_4}{(H_2)^2}$	6.0×10^{-10}	1.6×10^{-9}	3.2×10^{-9}
$2C + H_2 \rightleftarrows C_2H_2$ Carbon Hydrogen Acetylene	−58100	1 to 1	$K=\frac{C_2H_2}{H_2}$	1.1×10^{-13}	1.5×10^{-11}	5.7×10^{-10}
$6C + 3H_2 \rightleftarrows C_6H_6$ Carbon Hydrogen Benzene	−11300	3 to 1	$K=\frac{C_6H_6}{(H_2)^3}$	1.2×10^{-15}	1.7×10^{-15}	2.2×10^{-15}
$C_2H_6 \rightleftharpoons C_2H_4 + H_2$ Ethane Ethylene Hydrogen	−37900	1 to 2	$K=\frac{(C_2H_4)(H_2)}{C_2H_6}$	0.0027	0.094	1.28
$2CH_4 \rightleftharpoons C_2H_4 + 2H_2$ Methane Ethylene Hydrogen	−52400	2 to 3	$K=\frac{(C_2H_4)(H_2)^2)}{(CH_4)^2}$	0.0000001	0.00001	0.0004
$2CH_4 \rightleftharpoons C_2H_2 + 3H_2$ Methane Acetylene Hydrogen	−95900	2 to 4	$K=\frac{(C_2H_2)(H_2)^3}{(CH_4)^2}$	1.86×10^{-11}	0.0000001	0.00006
$C_2H_4 \rightleftharpoons C_2H_2 + H_2$ Ethylene Acetylene Hydrogen	−43500	1 to 2	$K=\frac{(C_2H_2)(H_2)}{C_2H_4}$	0.00018	0.0093	0.178
$C_2H_6 \rightleftharpoons C_2H_2 + 2H_2$ Ethane Acetylene Hydrogen	−81400	1 to 3	$K=\frac{(C_2H_2)(H_2)^2}{C_2H_6}$	0.0000005	0.00089	0.228
$C_4H_8 \rightleftharpoons 2C_2H_4$ Butylene Ethylene	−31800	1 to 2	$K=\frac{(C_2H_4)^2}{C_4H_8}$	1.41	31.6	258.0

[a] In various hydrocarbon reactions.

TABLE 4.—*Results of quantitative study of equilibria*—Continued.

GENERAL CRACKING REACTIONS.

Reaction.	Heat of reaction.	Volume change.	Equilibrium constant.	Value of constant.		
				K_{500}	K_{600}	K_{750}
$2C_{12}H_{26} \rightleftharpoons CH_4+C_{23}H_{48}$ Paraffin Paraffins	− 500	2 to 2	$K=\frac{(CH_4)(C_{23}H_{48})}{(C_{12}H_{26})^2}$	0.306	0.308	0.309
$2C_{12}H_{26} \rightleftharpoons C_{10}H_{22}+C_{14}H_{30}$ Paraffin Paraffins	000	2 to 2	$K=\frac{(C_{10}H_{22})(C_{14}H_{30})}{(C_{12}H_{26})^2}$	1.0	1.0	1.0
$C_{12}H_{26} \rightleftharpoons CH_4 + C_{11}H_{22}$ Paraffin Paraffin Olefin	− 16750	1 to 2	$K=\frac{(CH_4)(C_{11}H_{22})}{(C_{12}H_{26})}$	645	2820	15500
$C_{12}H_{26} \rightleftharpoons C_{10}H_{22} + C_2H_4$ Paraffin Paraffin Olefin	− 16100	1 to 2	$K=\frac{(C_{10}H_{22})(C_2H_4)}{(C_{12}H_{26})}$	1950	8300	43500
$C_{12}H_{26} \rightleftharpoons C_{12}H_{24} + H_2$ Paraffin Olefin Hydrogen	− 31800	1 to 2	$K=\frac{(C_{12}H_{24})(H_2)}{(C_{12}H_{26})}$	.0044	.0616	1.02
$C_{12}H_{26} \rightleftharpoons 5C_2H_4 + C_2H_6$ Paraffin Olefin Paraffin	− 80400	1 to 6	$K=\frac{(C_2H_4)^5 (C_2H_6)}{(C_{12}H_{26})}$	1.25×10^{16}	1.58×10^{19}	6.3×10^{22}
$C_{12}H_{26} \rightleftharpoons 6C_2H_4 + H_2$ Paraffin Olefin Hydrogen	−111600	1 to 7	$K=\frac{(C_2H_4)^6 (C_2H_6)}{(C_{12}H_{26})}$	1.2×10^{14}	2.0×10^{18}	1.6×10^{22}
$C_{12}H_{26} \rightleftharpoons C_2H_2 + 2C_5H_{12}$ Paraffin Acetylene Paraffin	− 47600	1 to 3	$K=\frac{(C_2H_2)(C_5H_{12})^2}{(C_{12}H_{26})}$	7.1×10^{2}	4.0×10^{4}	3.9×10^{6}
$C_{12}H_{26} \rightleftharpoons C_2H_2 + 2C_4H_{10} + C_2H_4$ Paraffin Acetylene Paraffin Olefin	− 61100	1 to 4	$K=\frac{(C_2H_2)(C_4H_{10})^2(C_2H_4)}{(C_{12}H_{26})}$	7.4×10^{2}	1.4×10^{9}	6.2×10^{11}
$C_{12}H_{26} \rightleftharpoons 6C_2H_2 + 7H_2$ Paraffin Acetylene Hydrogen	−381000	1 to 13	$K=\frac{(C_2H_2)^6 (H_2)^7}{(C_{12}H_{26})}$	1.0×10^{-20}	5.0×10^{-7}	1.2×10^{9}
$C_{12}H_{26} \rightleftharpoons 5C_2H_2 + 6H_2 + C_2H_4$ Paraffin Acetylene Hydrogen Olefin	−336800	1 to 12	$K=\frac{(C_2H_2)^5 (H_2)^6 (C_2H_4)}{(C_{12}H_{26})}$	3.5×10^{-15}	7.1×10^{-3}	2.9×10^{11}
$C_{12}H_{26} \rightleftharpoons 3C_2H_2 + 4CH_4 + C_2H_4$ Paraffin Acetylene Paraffin Olefin	−154400	1 to 8	$K=\frac{(C_2H_2)^3 (CH_4)^4 (C_2H_4)}{(C_{12}H_{26})}$	1.1×10^{11}	5.6×10^{16}	2.4×10^{23}
$C_{12}H_{24} \rightleftharpoons C_2H_2 + C_{10}H_{22}$ Olefin Acetylene Paraffin	− 29300	1 to 2	$K=\frac{(C_2H_2)(C_{10}H_{22})}{(C_{12}H_{24})}$	0.89	10.2	162
$C_{12}H_{24} \rightleftharpoons 4C_2H_2 \rightleftharpoons 4CH_4$ Olefin Acetylene Paraffin	−167600	1 to 8	$K=\frac{(C_2H_2)^4 (CH_4)^4}{(C_{12}H_{24})}$	5.6×10^{7}	7.1×10^{13}	7.8×10^{8}
$3C_2H_2 \rightleftharpoons C_6H_6$ Acetylene Benzene	+130700	3 to 1	$K=\frac{(C_6H_6)}{(C_2H_2)^3}$	2.0×10^{20}	6.3×10^{15}	5.7×10^{1}
$2C_2H_2 + C_2H_4 \rightleftharpoons C_7H_8$ Acetylenes Toluene	+131900	3 to 1	$K=\frac{(C_7H_8)}{(C_2H_2)^2 (C_2H_4)}$	6.8×10^{20}	2.2×10^{10}	1.6×10^{11}

INTERAROMATIC REACTIONS.

Reaction.	Heat of reaction.	Volume change.	Equilibrium constant.	Value of constant.		
				K_{656}	K_{725}	K_{800}
$4C_{10}H_{14} \rightleftharpoons 5C_8H_{10} + 3H_2$ Cymene, Xylene Hydrogen	−19000	4 to 8	$K = \frac{(C_8H_{10})^5(H_2)^3}{(C_{10}H_{14})^4}$	6.8×10^{29}	4.7×10^{31}	4.7×10^{33}
$7C_{10}H_{14} \rightleftharpoons 10C_7H_8 + 9H_2$ Cymene Toluene Hydrogen	−82000	7 to 19	$K = \frac{(C_7H_8)^{10}(H_2)^9}{(C_{10}H_{14})^7}$	2.0×10^{66}	1.4×10^{68}	2.0×10^{70}
$3C_{10}H_{14} \rightleftharpoons 5C_6H_6 + 6H_2$ Cymene Benzene Hydrogen	−68500	3 to 11	$K = \frac{(C_6H_6)^5(H_2)^6}{(C_{10}H_{14})^3}$	7.8×10^{40}	2.8×10^{42}	1.0×10^{44}
$7C_8H_{10} \rightleftharpoons 8C_7H_8 + 3H_2$ Xylene Toluene Hydrogen	−90800	7 to 11	$K = \frac{(C_7H_8)^8(H_2)^3}{(C_8H_{10})^7}$	1.1×10^{7}	6.6×10^{8}	3.1×10^{10}
$3C_8H_{10} \rightleftharpoons 4C_6H_6 + 3H_2$ Xylene Benzene Hydrogen	−65600	3 to 7	$K = \frac{(C_6H_6)^4(H_2)^3}{(C_8H_{10})^3}$	1.1×10^{13}	2.1×10^{14}	4.3×10^{15}
$6C_7H_8 \rightleftharpoons 7C_6H_6 + 3H_2$ Toluene Benzene Hydrogen	−40700	6 to 10	$K = \frac{(C_6H_6)^7(H_2)^3}{(C_7H_8)^6}$	2.9×10^{17}	3.2×10^{18}	2.7×10^{19}

As previously stated, each equilibrium constant has a definite value for definite temperature, but the reaction that proceeds at one temperature may be reversed at another temperature. Although pressure does not change the value of the equilibrium constant at a given temperature, it does have a material effect on the yields obtainable and the character of the product. The relation of temperature to pressure in the processes herein outlined, however, is essentially different from their relationship in any two-phase system. As regards processes carried out entirely in the gaseous state, the temperature can be chosen that is most favorable for the maximum of conversion and the minimum of decomposition into undesirable end products, whereas that pressure is employed which experiment has determined to be most favorable in increasing yields and improving the character of the product. This judicious selection of a combination of most favorable temperature and pressure is possible only in a single-phase system, for the reason fully outlined in the discussion of the limitations of two-phase systems.

TEMPERATURES EMPLOYED IN BUREAU OF MINES PROCESSES.

Gaseous-phase processes have been operated by the authors at temperatures higher than 950° C. and lower than 400° C. At the high temperatures, as was to be expected, the products went entirely to carbon and hydrogen and other fixed gases. The lower temperatures gave such slight conversion that their use was demonstrated to be commercially impracticable on account of the time element.

Gasoline formation seems to begin at a temperature higher than 400° C., and to approach a maximum between 500° and 550° C.

As regards aromatic hydrocarbons, it appears that a maximum content is discovered in oils cracked at a temperature of 650° to 700° C. It appears, however, that greater yields, on the basis of original oil, may be obtained at lower temperatures, as the lower temperatures permit a larger recovery of cracked oil. For practical operation, a temperature of 625° to 675° C. has been found most desirable. The particular temperature chosen will depend largely on the aromatic hydrocarbon desired in greatest quantity, as the yield of any given aromatic compound can be increased or diminished by approaching or departing from the temperature most favorable for its formation. For example, the proportionate yields of benzene and toluene can be varied at will up to the maximum percentage for each.

The temperatures indicated are relative only, as it has been found that each oil has its own optimum conditions. The suitability of a given temperature is dependent also on the size of the reaction chamber and the rate of feed of oil into the chamber.

PRESSURE.

According to an application of the principle of Le Chatelier, pressure favors any reaction resulting in decrease of volume and opposes any reaction that is accompanied by an increase in volume. It appears possible, therefore, to obtain indications of the effect of pressure by the simple process of writing an equation and summing up the volume changes.

The effect of varying the pressure when a hydrocarbon mixture is to be cracked may be illustrated by considering the general reaction $A = 2B$, in which a condition of equilibrium exists when $K = \frac{(P_B)^2}{P_A}$ and $KP_A = (P_B)^2$. This equilibrium constant is identical with the one mentioned under the discussion of temperature, but deals with the partial pressures at equilibrium under different conditions of pressure. If the value of K be assumed as equal to 1, then, for partial pressures, when A equals 100, B equals 10, or when A equals 0.01, B equals 0.1. In the first instance the partial pressure A is 10 times as great as that of B. In the second instance the partial pressure of A is only one-tenth the partial pressure of B. In other words, simply by changing the total pressure on the system and keeping all other pressures constant the ratio of A to B for the pressure chosen has been divided by 100. It follows that, as previously indicated, diminished pressure increases decomposition, whereas increase of pressure decreases decomposition. As illustrative of the practical bearing of these facts, it has been demonstrated that diminished pressure favors the manufacture of gas, whereas the manufacture of gasoline from heavy hydrocarbons is favored by increased pressure.

On first thought this statement with respect to gasoline formation may appear to be an anomaly. One would expect to use vacuum in order to help pull the larger molecules apart; but in gasoline formation it is important to produce all the saturated molecules possible, and this involves a building up or saturating (hydrogenation) process. Temperature accomplishes the dissociating of the big molecules and pressure causes saturation (hydrogenation) of the lower boiling and more stable gasoline molecules. The possibility of using high pressures, irrespective of the temperature, constitutes one of the most important advantages of the processes under discussion.

LIMITATIONS AS TO USE OF PRESSURE.

The velocity with which the reaction occurs is an important factor in determining the yield of any product when the time is fixed. According to the law of mass action, reaction velocity is proportional to concentration,[a] and, in gaseous-phase reactions, concentration is directly proportional to pressure. It appears, therefore, that increased pressure may be favorable to a point where the reaction proceeds with sufficient rapidity to attain a state of equilibrium. Increase in pressure above this point will tend to shift the equilibrium unfavorably if the reaction is one that is accompanied by increase of volume. It would seem to follow, therefore, that for each temperature there is some optimum pressure if the rate of feed is constant. For the lower temperatures, this pressure will be high; for high temperatures, it may be low.

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[a] The concentration of any substance in a gas mixture is proportional to its partial pressure and these terms are used interchangeably with suitable constants.

OBJECT OF HIGH PRESSURES.

The purpose of employing high pressure in the vapor phase is independent of any influence that the high pressure may have on the boiling points of hydrocarbons cracked. The process is not one of distillation, and boiling points are of no importance in the actual course of the reaction, as may be appreciated by considering the type reaction:

$$x\mathrm{A}=y\mathrm{B}-z\mathrm{C}$$

$$\text{Constant}=\frac{(\text{Concentration B})^{y}\ (\text{concentration C})^{z}}{(\text{concentration A})^{x}}$$

in which xA represents the high boiling hydrocarbons to be converted into lower boiling hydrocarbons, yB represents the lower boiling gasoline hydrocarbons, and zC represents hydrogen and the noncondensable hydrocarbons, such as methane (CH_4), ethane (C_2H_6), ethylene (C_2H_4), etc. Because of the tendency toward equilibrium in the system, it is evident that if the end products represented by zC are kept in the sphere of reaction the tendency is for xA hydrocarbons, representing the raw hydrocarbon material used, to form yB hydrocarbons (lower boiling hydrocarbons) in order to satisfy equilibrium. The greater the concentration of zC, the more pronounced will be the tendency of xA to form yB, and not zC.

In the conventional cracking processes zC constituents, being of lighter weight than the liquids, constantly pass up and out of the sphere of reaction, whereas an important feature of the present processes is the retention of the zC constituents in the sphere of reaction through control of the discharge of the evolved gases. If small narrow tubes are used, such as those mentioned previously in the review of the cracking processes, the end products represented by zC are necessarily swept out of the reaction chamber, as they can not be held therein. In processes of this character there is, necessarily, a great waste of valuable hydrocarbon material, as the tendency of all the hydrocarbon constituents present in the system to go into equilibrium causes a considerable part of the hydrocarbons to be continuously decomposed into end products. This decomposition is increased in proportion to increase of temperature.

INFLUENCE OF TIME IN VAPOR-PHASE CRACKING REACTIONS.

Time or duration of reaction is of high importance in the cracking or alteration of heavy oils into lighter products. The majority of organic reactions require time for their completion. In the present processes, the time factor may be controlled in various ways. The cracking apparatus may be lengthened, or the rate at which the oil is fed into the reaction chamber may be varied. It may be further effected by the employment of catalytic agents. The effect of a catalyzer is to accelerate the reaction.

The time factor is also intimately related to the temperatures and pressures employed. It is obvious that if a high temperature be employed, a lesser time is required than if a relatively lower temperature be used. A slow rate of feeding oil and a relatively low temperature will accomplish practically the same results as a relatively faster rate of feed and a much higher temperature, with the single difference that more time will be necessary.

In the processes under consideration it is preferable to maintain a given rate of feed for the favorable conditions of temperature and pressure established by experimentation for any particular oil. For example, the following conditions existed in the 8-inch tubes used in the commercial production of benzene and toluene from a fuel-oil distillate. With a feed of approximately 15 gallons per hour, temperatures of 650° to 675° C., and a pressure of 150 pounds per square inch, the results are satisfactory. If the temperature is increased to 750° C., a rate of feed of between 25 and 30 gallons per hour would be necessary in order to obtain similar results. The mechanical difficulties introduced by the higher temperatures, however, decrease their feasibility in commercial work. In the production of gasoline from high-boiling distillates, the rate of feed has been as high as 70 gallons per hour, under a pressure of 200 pounds per square inch, and a temperature of 600° C.

SPEED OF REACTION IN VAPOR-PHASE PROCESS.

The introduction of a small volume of petroleum into a highly heated chamber has a number of advantages as regards efficiency. All of the material is heated immediately and uniformly. At the same time the danger incident to blowouts or to receptacles giving way is much less than in a process in which large quantities of oil are heated cracking temperatures.

Likewise, as all the material to be treated at a given time is present in the reaction chamber in a gaseous form, and as all the constituents of an equilibrium system are present, it is possible for new hydrocarbon formations to occur almost simultaneously with the breaking up of the heavier gasified hydrocarbons.

EFFECT OF CONTACT SURFACE.

The factor of contact surface is of secondary importance in the processes under consideration, except as it may be associated with catalytic effects. This factor is of importance, however, in liquid-phase cracking processes, as in them much of the cracking is due to local superheating of oil in contact with the walls of the container. The higher temperatures possible in the vapor phase bring about the same acceleration in reaction velocity that is accomplished by the use of catalyzers.

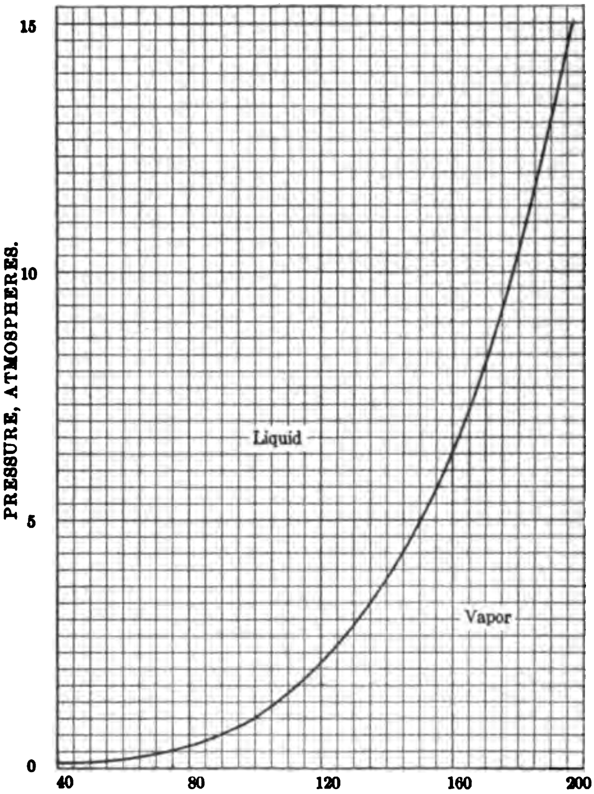

FIGURE 8.—Vapor-pressure curve for water.

ADVANTAGES OF SINGLE-PHASE CRACKING PROCESS OVER TWO-PHASE PROCESSES.

The advantages of the one-phase system become evident on consideration of some principles of physical chemistry. In a two-phase system temperature and pressure are functions of each other; in a one-phase system they are mutually independent. The simple vapor-pressure curve for water shown in figure 8 makes this clear. If liquid and vapor are both present equilibrium is represented by a line, and any deviation from the combination of conditions indicated by this line results in the disappearance of a phase. Thus if water is heated to a temperature higher than 100° C. under atmospheric pressure it is entirely changed into vapor. But water vapor will remain as such under any desired condition of temperature and pressure within the limits that do not cause condensation of liquid.

Of course, petroleum is not exactly comparable with water, as petroleum is not a single compound but a complex mixture. A certain limited degree of flexibility of control of the two variables, temperature and pressure, is possible even in a two-phase petroleum system, as equilibrium is maintained by changes in the relative composition of gaseous and liquid phases. But beyond a certain point there is no flexibility, and, in actual fact, cracking conditions in all two-phase processes are in large degree controlled by the nature of the oil used as original material.

A possible objection to the vapor-phase processes herein discussed might be that when these processes are applied to an oil with constituents having boiling points as high as 550° C., and the temperature in the tube, for example, 500° C., all the hydrocarbons are not vaporized. However, all the oil acted on is in the gaseous phase. Any heavier ungasified hydrocarbons will fall instantly out of the sphere of reaction. The cracking tubes are vertical and the force of gravitation attends to any deficiencies in temperature. However, when high-boiling residues are being treated it is perfectly practicable to increase correspondingly the temperature in the tube. When the hydrocarbons under treatment boil at a temperature below that of the tube there is little difficulty in obtaining complete vaporization, provided a suitable rate of flow is maintained. Moreover, as the processes are so arranged that cracking takes place in the gaseous phase, the temperature and the pressure may be controlled independently.

As pressure and temperature are the two most important chemical factors involved in any gaseous chemical process, the importance of being able to change either one of these variables at will without varying the other is readily seen. Moreover, the mode of treatment used in the processes herein described is in no way limited to the petroleum industry. Any reactions possible between substances capable of vaporization may be conducted in a single (gaseous) phase. Such a procedure has two decided advantages, as follows:

1. The high speed of reaction incident to high temperatures.
2. Thorough mixing and contact.

It is believed that the time will soon come when it will be found advantageous to conduct in the gaseous phase many reactions that now take place in the liquid phase.

OBJECTIONS TO USE OF TUBES.

A characteristic feature of many cracking processes is the use of tubes of various lengths, sizes, and arrangements in place of the conventional still. The following objections are typical of those generally raised:

If large tubes are used, it is claimed that there is a tendency to short circuiting or channeling, much of the original material being thus permitted to pass through without attaining the desired temperature. Attempts to apply higher temperatures result in uneven heating, the periphery of the tube being hotter than the interior. The average amount of heat supplied may be thus brought to the desired quantity, but some of the oil is cracked at high temperatures and some at low. As a result the desired products may never be obtained, for their properties are in no way supplied by mixing the compounds produced by cracking at higher with those produced at lower temperatures. As is shown subsequently, all disadvantages of this sort may be eliminated by simple mechanical improvements.

A more serious objection to cracking in tubes, particularly in those of small internal diameter, such as the $\frac{3}{16}$-inch or $\frac{1}{2}$-inch tubes employed in some of the processes previously mentioned, is the deposition of carbon. This can not be avoided when hydrocarbon mixtures are cracked, as the temperature necessary to bring about partial decomposition of the lighter molecules will inevitably bring about some total decomposition.

Obviously the difficulty of removing from a long continuous coil a deposit of accumulating carbon is considerable. Attempts have been made to minimize carbon deposition by maintaining a high rate of flow through the tubes. In Hall's process, for example, a speed equivalent to 6,000 feet per minute is attained, on the theory that carbon formation and accumulation is much more pronounced at a slow rate of flow than at one of great rapidity.

It is obvious that a coating of carbon on the walls reduces the thermal efficiency of the tube, thereby necessitating a greater fuel consumption to maintain the desired temperature. The carbon deposition necessarily incident to high temperature, with consequent plugging of small tubes, has always justly been considered an objection to any tubular system.

Difficulties on account of the adherence of carbon to the heated contact surface are not confined to tubes. Humphreys,[a] in his letters patent for an improved form of still, states that in the original Burton process only about one-third of the initial charge of oil could be distilled before difficulties resulted through carbon accumulating on the still bottom. Humphreys has endeavored to obviate the danger resulting from excessive carbon deposition by having a still equipped with one or more false bottoms.

ADVANTAGES OF USING LARGE TUBES.

The use of tubes of considerable diameter in place of small tubes obviates the objections advanced against the use of tubes and gives a high degree of efficiency. With vertical tubes 8 inches or more in diameter it has been possible to remove carbon continuously by a mechanical device to be described.

The carbon deposition incident to the gasoline process is almost negligible.

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[a] Humphreys, R. E., U. S. Patent 1122003, Dec. 22, 1914.

In the benzene-toluene process, carbon formation is unavoidable. Fortunately, however, it has been possible so to regulate furnace conditions that the carbon appears either as a light powder or a tarry sludge, rather than as a dense and adherent coke.

The use of tubes gives a number of advantages lacking with the conventional still. First, the vertical arrangement of the tubes permits carbon to pass down and out of the cracking or reaction zone, so that it does not interfere with the control of furnace conditions. Second, the cheapness of tubes as compared with stills is an important recommendation. Third, the danger incident to the treatment of large quantities of liquid at a given time is minimized, this feature alone being sufficient to justify the use of the tubes. Fourth, continuous 24-hour-a-day operation is possible with tubes, whereas with the conventional types of still, operation is intermittent because of the necessity of stopping frequently in order to remove unconverted liquid and carbon deposits from the still.

As regards efficiency and low initial cost the tube is believed to have decided advantages over the large receptacle or retort. The fact remains, however, that in the most extensively employed cracking process for producing gasoline (the Burton-Humphreys) stills are used, and millions of gallons of oil are handled yearly. This is, perhaps, evidence of the skill and ability of the inventors and of the operating company rather than proof of the intrinsic superiority of the still.

LABORATORY EXPERIMENTS LEADING TO DISCOVERY OF BUREAU OF MINES PROCESSES.

DESCRIPTION OF EXPERIMENTAL APPARATUS.

The apparatus used for the laboratory experiments here described is shown in figures 9 and 10. It consisted essentially of an electrically heated furnace body made of wrought-iron pipe 1½ inches in diameter and 32 inches long. For a distance of 18 inches the pipe was wrapped with nichrome resistance wire, an insulation of asbestos being placed between the iron pipe and the resistance wire. A ¾-inch wrought-iron pipe was welded at right angles into the furnace body to serve as a container for the pyrometer point. Both the furnace body and the side tube were incased in a 5-inch asbestos pipe covering to minimize radiation.

In the upper part of the furnace tube a perforated plate was placed, upheld by a thin rod running vertically through the furnace. The space above the plate was filled with steel balls to facilitate the vaporization of the oil entering the tube, the steel balls being purposely kept above the cracking zone of the furnace in order to preserve their intended function as a vaporizer.

At the upper end of the furnace a sight-feed oil cup of approximately 1-quart capacity was used. In order to permit the oil to flow into the tube, the pressure in the furnace and that in the oil cup were equalized through a small tube communicating with the furnace body below the point of entrance of the oil feed. As a result, regard-

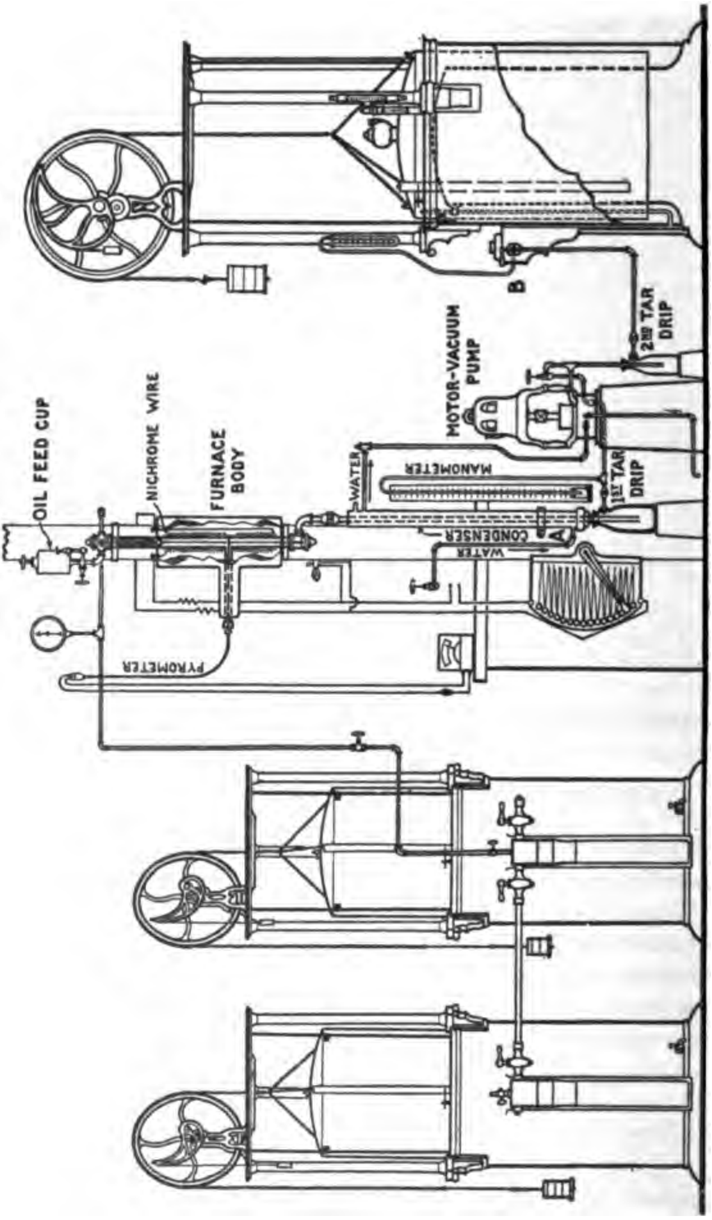

FIGURE 9.—Experimental furnace used in laboratory for cracking oils in vapor phase, arranged for operating at atmospheric or diminished pressure. Left to right, admixture gas tanks, oil-cracking machine, gas collector. A, condenser, with glass tar drip attached; B, gas connection to tank.

less of the operative pressure, the oil was always under a pressure equal to its head.

The gaseous products were discharged from the furnace through a Liebig condenser A into a tar drip, which for atmospheric or diminished pressure consisted of a thick glass bottle. The continuing tube carried the fixed gases to a gasometer, where the volume of gases generated could be measured. The apparatus was also connected with a rotary type vacuum pump when pressures below atmospheric were used. By means of a by-pass connection joining the outlet and the inlet of the pump the valve could be so regulated as to maintain any desired vacuum down to one-thirtieth of an atmosphere. When pressures higher than atmospheric were employed, a metal tar collector, a connecting pipe, and a pressure-release valve were used. The gas generated in the furnace body was used to create the pressure on the system, which was controlled by the release valve.

The electric-heating equipment of the furnace body permitted the accurate regulation of temperature up to 1000° C., the temperature measurements being made by means of a thermocouple inserted in the interior of the furnace.

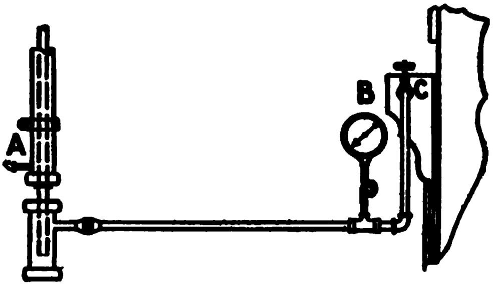

FIGURE 10.—Details of pressure connections of apparatus shown in figure 9. A, lower part of condenser, with steel tar collector attached; B, pressure gage; C, pressure-release valve.

PROCEDURE IN MAKING A RUN OR EXPERIMENT.

The apparatus under consideration is operated substantially as follows:

By passing an electric current through the resistance wire the reaction chamber of the tube is heated to the temperature desired for the material to be cracked. When the tube and the small metal balls constituting the filling material have attained the appropriate temperature, the valve at the bottom of the oil-feed cup is opened, and the material is allowed to flow by gravity into the upper part of the tube at a predetermined rate, a certain number of drops per minute. The valve at the lower end of the apparatus is closed in order to bring the concentration of permanent gases to a desired point. After the pressure has built up the valve can be opened from time to time to release the excess pressure in the cracking tube. Hydrocarbon gases pass from the upper part of the tube, which constitutes the gasification zone, downward into the cracking zone where cracking and recombination of the molecules takes place. After having passed through the cracking zone, the reaction products are condensed and collected in a suitable receptacle, the rate of flow from the reaction or cracking zone being regulated by the pressure-release valve.

In the conduct of the experiments here described 400 to 600 grams of oil were run through the furnace. After the completion of the run the quantity of resulting oil was determined, and the deposited carbon was scraped out of the furnace and weighed. The evolved gas was collected in a gas holder and its volume measured and recorded. Samples of evolved gases were removed from the gas holder and analyzed.

DISTILLATIONS MADE.

Distillations of the recovered oils were made in 250-c. c. flasks. The neck of each was five-eighths of an inch in diameter and 6 inches long, and fitted with a 5-inch column of aluminum beads which produced a still head of the Hempel type. Charges of 200 grams were distilled, and cuts made at temperatures of 150° C. and higher at intervals of 50° C. The type of apparatus used is more fully described in a recent publication by two of the authors.[a]

DETERMINATION OF SPECIFIC GRAVITY AND VISCOSITY.

Specific gravities of the original oils and distillation cuts of recovered oils were determined by means of a Westphal balance with a plummet of 1 c. c. displacement, or else with Drushel-type pycnometers.[b]

Viscosities for original and recovered oils were measured by the Engler viscosimeter, and the results expressed in Engler degrees, which represent the ratios between the rates of flow of oils and of water.

Coincident with the conversion experiments a study was made of the relations among the physical constants of the petroleum used. The results of these studies have been already presented.[c]

MAIN FEATURES OF EXPERIMENTS.

The main features of the experiments may be summarized as follows:

"RUN" OR MAJOR EXPERIMENT.

1. Cracking of 400 to 600 grams of oil in furnace at regulated temperature and pressure.
2. Determination of quantities of oil, gas, and carbon resulting.

SUPPLEMENTARY DETERMINATIONS.

1. Distillation of (*a*) uncracked oil, and (*b*) cracked oil.
2. Determination of specific gravities of uncracked oil, cracked oil, and individual distillation cuts.

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[a] Rittman, W. F., and Dean, E. W., Analytical distillation of petroleum: Jour. Ind. and Eng. Chem., vol. 7, March and September, 1915, p. 185-195, 754-760.

[b] Rittman, W. F., and Dean, E. W., Loc. cit.

[c] Rittman, W. F., and Egloff, Gustav, Relations among the physical constants of petroleum distillates: Jour. Ind. and Eng. Chem., vol. 7, July, 1915, p. 578, 582.

3. Determination of viscosity and refractive index.
4. Chemical tests of distillates.
5. Analyses of evolved gases.

RESULTS OF EXPERIMENTS.

The results of the experiments conducted are given in Tables 5, 6, 7, 8, and 9. Because of the great variety of data presented, the results for each individual oil have been incorporated in separate tables.

Most attention was given the three United States oils, namely, a Pennsylvania refined burning oil, representing the paraffin oils of the Appalachian oil fields, an Oklahoma fuel oil, representing the mixed-base oils of the Mid-Continent field, and a Kern River crude oil, typifying the oils of asphaltic base obtained from California. Sufficient data were obtained for the Mexican and Russian oils to show that in spite of differences in physical properties and chemical constitution results of the same general character could be obtained as with petroleum from the United States. All of the oils tested were suitable for experimental purposes by reason of their freedom from low-boiling hydrocarbons.

The data reported here are only those that were generally available for all the oils used, and consequently do not show the entire range of temperatures and pressures embraced by the experiments, although they cover a wide range and embrace the conditions of commercial importance in the cracking reaction. Lower temperatures than those shown will not produce a decomposition rapid enough to be commercially feasible, whereas the extreme high temperatures produced a maximum of ultimate end products, namely, carbon and fixed gases. The range of pressures used was as great as can be managed in an apparatus of ordinary construction, in conjunction with high temperatures, whether in small or large scale operations. Tables 5 to 9 follow.

TABLE 5.—*Results of cracking Pennsylvania refined burning oil under various conditions of temperature and pressure.*

[Specific gravity of original oil, 0.802; viscosity, 1.05° Engler.]

Temperature, ° C	Original oil	850	750	750	750	750	600	600	600
Pressure, atmospheres		1	12	6	1	(a)	24	18	12
Oil used, grams		781	872	545	898	800	568	572	547
Oil recovered, per cent		9.7	15.9	16.9	16.5	33.9	37.3	40.5	43.6
Specific gravity of recovered oil.		1.026	.910	1.020	1.012	.905	.962	.926	.918
Viscosity of recovered oil, ° Engler.						1.04			1.11
Carbon, per cent		7.4	24.3	15.3	8.7	16.1	7.5	5.3	3.6
Gas, liters per kilogram of oil used.		817		620	612				240
Illuminants in gas, per cent		12.3		10.8	32.0				19.6
Calorific value of gas, B. t. u. per cubic foot.		822		1,058	1,239				1,406

[a] Vacuum.

RESULTS OF DISTILLATION.

First drop at (° C.)	120		70		75		70		60				36		30		33	
Temperature.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.
° *C.*																		
100			18.0	0.875	30.2	0.865	31.6	0.876	16.8	0.876	3.7	0.800	19.0	0.837	19.0	0.816	18.0	0.807
150	4.8	0.763	7.0	.859	9.4	.834	9.7	.860	14.3	.878	14.3	.832	21.5	.862	25.0	.855	24.2	.851
200	46.7	.784	10.0	.885	17.9	.824	8.3	.873	8.9	.922	39.2	.848	9.3	.876	12.7	.860	15.0	.857
250	39.0	.811	30.0	Solid.	25.5	Solid.	22.2	Solid.	20.7	Solid.	23.7	.904	13.5	Solid.	15.3	.931	14.0	.930
300									8.2	Viscous at 21°.	6.3	.956						
Total to 150	4.8	.763	25.0	.873	39.6	.858	41.3	.872	31.1	.877	18.0	.826	40.5	.847	44.0	.837	42.2	.833
Total 150 to 250	85.7	.797	40.0		43.4		30.5		29.6		62.9	.858	22.8		28.0	.900	29.0	.893

Temperature, ° C.	600	600	600	500	500	500	500	500	500	450
Pressure, atmospheres	6	1	(a)	24	18	12	6	1	(a)	12
Oil used, grams	861	591	750	576	539	561	951	750	750	575
Oil recovered, per cent	31.4	59.9	97.4	72.5	79.7	84.2	57.8	94.5	...	93.1
Specific gravity of recovered oil	.885	.840	.811	.828	.819	.808	.810	.799	.802	.809
Viscosity of recovered oil, ° Engler	1.00	...	1.04	.97	.96	.98	1.00	1.00	1.04	1.02
Carbon, per cent	2.4	0.6	0.0	1.2	0.0	0.0	0.0	0.0	6 g(?)	5.1
Gas, liters per kilogram of oil used	...	...	16.7	...	...	...	53.8	...	5.3	...
Illuminants in gas, per cent	...	...	54.5	...	...	...	26.1	52.9	54.9	...
Calorific value of gas, B. t. u., per cubic foot	...	...	1,441	...	...	...	1,192	1.458	1.333	...

a Vacuum.

RESULTS OF DISTILLATION.

First drop at (° C.)	...		31		...		32		30		30		40		36		...		30	
Temperature.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.
° C.																				
100	14.5	0.772	8.7	0.759	0.5	...	13.5	0.726	9.5	0.714	9.5	0.725	5.0	0.735	1.0	0.718	0.0	...	3.5	0.715
150	25.0	.829	18.5	.803	3.5	0.770	16.2	.802	14.5	.790	14.3	.786	10.0	.780	6.7	.761	3.0	0.760	8.3	.778
200	21.0	.838	36.5	.815	44.5	.791	27.3	.813	32.7	.806	34.0	.801	38.8	.793	42.5	.787	46.0	.786	34.0	.793
250	16.5	.889	23.5	.850	41.0	.818	25.0	.850	28.5	.839	27.3	.831	33.0	.820	38.0	.814	37.5	.813	33.0	.820
300	7.5	.956	5.7	.901	...	...	...	...	...	...	...	...	8.0	.851	...	...	...	...	...	...
Total to 150	39.5	.809	27.2	.790	4.0	.769	29.7	.771	24.0	.765	23.8	.763	15.0	.767	7.7	.758	3.0	.760	11.8	.764
Total 150 to 250	37.5	.861	60.0	.827	85.5	.804	52.3	.832	61.2	.818	61.3	.814	71.8	.804	80.5	.800	83.5	.798	67.0	.805

TABLE 6.—*Results of cracking Oklahoma fuel-oil residuum under various conditions of temperature and pressure.*

[Specific gravity of original oil, 0.894; viscosity, 35.40° Engler.]

Temperature, ° C	Original oil	850	800	750	750	750	750	650	600	600	600	600
Pressure, atmospheres	....	(a)	(a)	12	6	1	(a)	(a)	24	18	12	6
Oil used, grams	....	862	937	596	703	1,325	912	1,159	581	574	554	812
Oil recovered, per cent	....	23.1	29.3	20.2	22.2	17.5	51.7	88.7	83.9	35.6	62.2	87.3
Specific gravity of recovered oil.	....	.997	.963	.965	1.040	1.039	.925	....	1.008	.978	.907	.981
Viscosity of recovered oil, ° Engler.	....	....	1.64	....	....	....	1.44	....	....	....	1.48	1.06
Carbon, per cent	....	5.8	4.1	24.6	20.0	11.3	4.1	2.2	17.2	13.2	6.7	9.6
Gas, liters per kilogram of oil used.	....	570	....	....	....	597	315	53.7	....	393	265	356
Illuminants in gas, per cent.	....	49.8	58.7	....	....	28.1	64.9	58.9	....	10.3	21.1	26.1
Calorific value of gas, B. t. u., per cubic foot.	....	1,373	1,486	....	....	993	1,580	1,584	....	....	1,459	1,390

a Vacuum.

RESULTS OF DISTILLATION.

First drop at (° C.)	....		70		46		50		....		65		38		62		37		35		30		32	
Temperature.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.
° C.																								
100	0.0	......	8.9	0.873	6.5	0.827	34.4	0.858	19.6	0.879	13.7	0.873	5.3	0.775	1.2	......	21.0	0.845	24.0	0.833	10.0	0.785	16.5	0.805
150	.3	......	9.7	.868	13.0	.856	9.6	.853	7.1	.857	13.4	.876	9.3	.827	2.5	0.798	17.4	.871	18.0	.867	9.8	.839	21.7	.850
200	2.0	0.801	6.8	.921	7.5	.872	8.3	.855	5.8	.850	5.1	.917	9.0	.849	4.0	.826	4.5	.900	5.7	.891	5.0	.848	12.3	.853
250	5.5	.826	18.9	......	11.2	.919	18.7	Solid.	19.2	Solid.	19.1	Solid.	10.7	.879	11.0	.848	11.8	.967	10.8	.977	11.2	.873	16.0	.902
300	14.0	.841	......	......	10.8	.928	......	......	......	......	6.9	.994	12.5	.893	15.5	.855	......	......	......	......	14.0	.885	8.0	.956
Total to 150	.3	......	18.6	.870	19.5	.847	44.0	.857	26.7	.874	27.1	.875	14.8	.811	3.7	.793	38.4	.855	42.0	.853	19.8	.813	38.2	.832
Total 150 to 250	7.5	.819	25.7	......	18.7	.899	27.0	......	25.0	......	24.2	......	19.7	.852	15.0	.842	16.3	.963	16.5	.948	16.2	.866	28.3	......

Temperature, ° C.	600	600	550	550	500	500	500	500	500	450	450
Pressure, atmospheres	1	(a)	24	18	24	12	6	1	(a)	18	12
Oil used, grams	982	1,692	577	570	590	518	601	705	536	550	547
Oil recovered, per cent	50.8	100.0	38.1	55.0	63.8	88.2	91.8	99.0	99.8	76.9	87.7
Specific gravity of recovered oil	.906	.899	......	.908	.877	.870	.867	.872	.901	.882	.878
Viscosity of recovered oil, ° Engler	1.27	7.29	1.05	......	1.25	1.32	1.54	2.51	13.42	1.50	1.78
Carbon, per cent	3.1	0.0	9.2	7.0	4.6	1.4	1.2	0.0	0.0	0.0	0.0
Gas, liters per kilogram of oil used	289	38.1	311	......	......	......	53.5	88.0	......	......	......
Illuminants in gas, per cent	50.4	61.2	14.9	......	......	......	15.7	30.6	......	......	......
Calorific value of gas, B. t. u., per cubic foot	1,532	1,587	1,355	......	......	......	1,279	1,236	......	......	......

[a] Vacuum.

RESULTS OF DISTILLATION.

First drop at (° C.)	38		66		30		40		30		30		33		47		100		30		35	
Temperature.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.
° *C.*																						
100	10.3	0.769	1.0	......	17.5	0.758	16.3	0.752	13.0	0.718	9.3	0.712	6.2	0.704	2.3	0.724	0.0	......	6.2	0.748	5.0	0.713
150	15.0	.817	1.8	0.791	15.5	.842	15.5	.833	9.3	.803	6.7	.790	6.0	.777	2.7	.766	0.8	......	6.3	.813	5.3	.783
200	10.5	.840	3.7	.817	9.3	.865	7.5	.859	9.0	.827	7.5	.821	5.8	.810	4.2	.802	2.2	0.806	4.5	.827	5.5	.811
250	11.2	.872	9.5	.839	11.0	.917	11.0	.905	12.5	.863	10.7	.853	9.7	.840	11.8	.835	7.0	.829	8.8	.856	9.7	.839
300	14.0	.894	13.0	.850	......	......	......	......	14.5	.886	14.3	.868	16.0	.858	11.2	.848	13.5	.844	......	......	14.8	.855
Total to 150	25.3	.798	2.8	.791	33.0	.786	31.8	.792	22.3	.755	16.0	.752	12.2	.741	5.0	.752	0.8	......	12.5	.782	10.3	.755
Total, 150 to 250	21.7	.855	13.2	.827	20.3	.893	18.5	.887	21.5	.847	18.2	.739	15.5	.828	16.0	.827	9.2	.824	13.3	.847	15.2	.829

TABLE 7.—*Results of cracking California (Kern River) crude oil under various conditions of temperature and pressure.*

[Specific gravity of original oil, 0.970; viscosity, 35.7° Engler.]

Temperature, ° C.	Original oil.	850	800	750	750	750	750	600	600	600
Pressure, atmospheres	......	(a)	(a)	12	6	1	(a)	24	18	12
Oil used, grams	......	646	582	315	539	603	578	569	517	597
Oil recovered, per cent	......	34.2	43.7	21.1	20.9	20.9	47.4	31.6	33.7	52.5
Specific gravity of recovered oil	......	1.025	1.017	1.022	1.025	1.092	.963	1.030	.997	.996
Viscosity of recovered oil, ° Engler	......	......	3.95	......	......	......	......	......	......	1.79
Carbon, per cent	......	7.1	6.9	38.8	36.2	9.9	8.7	26.7	27.5	19.3
Gas, liters per kilogram of oil used	......	429	......	......	556	495	104	......	385	248
Illuminants in gas, per cent	......	42.2	......	......	5.6	20.5	49.8	......	4.4	11.5
Calorific value of gas, B. t. u., per cubic foot	......	1,296	......	......	1,004	875	1,464	......	808	1,241

a Vacuum.

RESULTS OF DISTILLATION.

First drop at (° C.)	......		......		......		70		67		70		......		40		33		67	
Temperature.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.
° *C.*																				
100	......	......	8.0	0.864	12.5	0.849	32.8	0.852	31.1	0.879	13.4	0.870	1.7	......	11.3	0.837	21.3	0.847	11.0	0.828
150	......	......	6.0	.861	7.5	.859	14.8	.839	8.4	.864	10.8	.875	6.8	0.814	22.7	.871	17.5	.870	11.5	.853
200	1.8	0.806	8.0	.879	8.3	.874	6.5	.862	2.6	.886	3.6	.917	7.1	.841	4.7	.908	5.3	.893	7.0	.870
250	5.5	.854	14.7	.953	14.0	.929	22.2	Solid.	11.1	Solid.	17.5	Solid.	10.8	.874	10.6	.990	13.1	.980	10.5	.922
300	10.0	.886	9.0	.962	10.0	.963	......	......	......	......	......	......	13.3	.906	......	......	......	......	14.0	.946
Total to 150	......	......	14.0	.863	20.0	.852	47.6	.848	39.5	.875	24.2	.873	8.5	.814	34.0	.862	38.8	.867	22.5	.842
Total 150 to 250	7.3	.841	22.7	.926	22.3	.908	28.7	......	13.7	......	21.1	......	17.9	.859	15.3	.964	18.4	.953	17.5	.903

Temperature, ° C.	600	600	600	550	500	500	500	500	500	450
Pressure, atmospheres	6	1	(a)	24	24	12	6	1	(a)	12
Oil used, grams	987	611	551	563	571	621	571	514	510	613
Oil recovered, per cent	43.9	46.6	81.8	40.6	53.6	64.8	71.5	91.8	73.6	75.4
Specific gravity of recovered oil	.984	.966	.960	.991	.942	.925	.928	.939	.950	.924
Viscosity of recovered oil, ° Engler	1.15	1.98	....	....	1.35	1.85	1.52	6.16	....	2.71
Carbon, per cent	16.2	12.3	13.6	21.1	11.7	14.5	10.1	3.7	11.9	2.8
Gas, liters per kilogram of oil used	314	205	33.2	275	....	....	103	34.2	75.2	....
Illuminants in gas, per cent	17.6	41.7	42.4	8.3	....	....	19.6	32.8	41.7	....
Calorific value of gas, B. t. u., per cubic foot	1,295	1,479	1,310	1,264	....	....	1,358	1,184	1,180	....

[a] Vacuum.

RESULTS OF DISTILLATION.

First drop at (° C.)	35		33		30		30		33		35		38		50		....		40	
Temperature.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.
° C.																				
100	16.5	0.826	8.3	0.767	3.3	0.800	12.5	0.787	12.8	0.736	11.0	0.729	8.5	0.759	3.2	0.727	0.3	....	4.3	0.720
150	19.2	.863	14.5	.831	3.2	.832	17.3	.862	12.3	.828	11.0	.812	10.7	.815	4.5	.785	1.2	0.807	5.2	.798
200	7.5	.887	8.0	.861	4.0	.847	8.2	.894	7.0	.863	8.3	.848	7.8	.858	5.3	.824	1.0	....	5.5	.825
250	10.8	.952	12.0	.902	11.3	.883	11.0	.966	12.3	.910	10.7	.891	9.5	.893	8.8	.865	5.3	.848	9.3	.867
300	10.0	.988	15.5	.937	9.7	.905	......	....	......	....	10.5	.920	11.0	.919	13.2	.891	12.0	.883	11.5	.894
Total to 150	35.7	.847	22.8	.808	6.5	.817	29.8	.829	25.1	.783	22.0	.776	19.2	.794	7.7	.765	1.5	....	9.5	.764
Total 150 to 250	18.3	.926	20.0	.884	15.3	.873	19.2	.936	19.3	.893	19.0	.872	17.3	.876	14.1	.848	6.3	.845	14.8	.851

TABLE 8.—*Results of cracking Mexican crude oil under various conditions of temperature and pressure.*

[Specific gravity of original oil, 0.986; viscosity, over 300° Engler.]

Temperature, °C	Original oil	750	600	600	600	500	500	400
Pressure, atmospheres		(a)	12	1	(a)	12	1	10 to 11
Oil used, grams		812	479	628	500	573	591	527
Oil recovered, per cent		37.3	47.2	40.2	66.0	54.5	72.8	73.5
Specific gravity of recovered oil		.992	.941	.992	.942	.918	.930	.934
Viscosity of recovered oil, ° Engler		2.70		1.96	6.73	1.15	1.88	4.43
Carbon, per cent		10.5	13.1	16.4	9.0	16.2	9.8	13.8
Gas, liters per kilogram of oil used		321	250	249.6		158	64	
Illuminants in gas, per cent		51.8		38.3	47.0	19.4	33.1	
Calorific value of gas, B. t. u., per cubic foot		1,411		1,510	1,487	1,491	1,366	

[a] Vacuum.

RESULTS OF DISTILLATION.

First drop at (°C.)					30		40		70		30		55		35	
Temperature.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.
°C.																
100	0.0		8.3	0.822	13.3	0.765	13.0	0.802	2.3	0.751	13.5	0.721	7.0	0.739	4.8	0.729
150	1.1	0.759	5.0	.836	12.5	.829	8.3	.837	3.5	.782	13.3	.807	8.7	.792	6.0	.789
200	2.5	.798	7.7	.868	8.0	.846	9.2	.860	8.3	.811	11.0	.837	8.5	.825	7.0	.816
250	8.3	.843	10.5	.915	11.0	.899	13.0	.923	10.5	.846	13.0	.884	12.5	.875	11.0	.856
300	25.2	.871	14.5	.941	11.7	.943	12.5	.968	15.5	.880	12.2	.945	11.7	.902	13.7	.898
Total to 150	1.1	.759	13.3	.827	25.8	.797	21.3	.816	5.8	.771	26.8	.766	15.7	.770	10.8	.762
Total, 150 to 250	10.8	.833	18.2	.897	19.0	.877	22.2	.909	18.8	.829	24.0	.862	21.0	.854	18.0	.843

TABLE 9.—*Results of cracking Russian oil under various conditions of temperature and pressure.*

[Specific gravity of original oil, 0.897; viscosity 11.92° Engler.]

Temperature, °C.	Original oil	675	500
Pressure, atmospheres		12	12
Oil used, grams		443	387
Oil recovered, per cent		28.3	77.3
Specific gravity of recovered oil		.965	.880
Viscosity of recovered oil, ° Engler			1.44
Carbon, per cent		21.7	0.0
Gas, liters per kilogram of oil used		506	.....
Illuminants in gas, per cent		10.4	19.5
Calorific value of gas, B. t. u., per cubic foot		1,472	1,730

RESULTS OF DISTILLATION.

First drop at ° C.			40		30	
Temperature.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.
°C.						
100	0.0		30.5	0.863	7.3	0.718
150	0.0		16.1	.867	7.8	.796
200	1.5		4.7	.891	5.7	.829
250	10.7	0.836	13.9	Solid.	13.8	.856
300	16.3	.859	5.9	.964	16.5	.881
Total to 150	0.0		46.6	.865	15.1	.761
Total 150 to 250	12.2	.836	18.6		19.5	.846

The features of most importance in the results presented in the foregoing tables are discussed under their appropriate heads.

OIL RECOVERED.

The relative quantity of original oil recovered is a factor of the highest commercial importance in any cracking process. The conditions under which the experiments were conducted were such as to prevent an accurate determination of the yields of recovered oil, especially when the experiments were conducted under pressure. In such tests, the pressure was built up by the gases created by the cracking of the starting material. In order to obtain the pressure desired, a considerable quantity of the charge of oil to be treated was consumed, as it had to be completely decomposed in the building up of the requisite pressure, so that the quantity of oil recovered was materially reduced.

When the furnace was operated at the lower temperatures, the furnace body had to be superheated at the start, in order to raise the pressure in the reaction chamber to the desired mark before too much of the original material had been run through. The effect of this procedure on the recovery of oil may be gathered from the following example:

If a run at 500° C. and 18 atmospheres was desired, it was necessary to start the furnace at 750° C. and at atmospheric pressure and to run in a quantity of oil until the pressure reached the desired height, after which the temperature was allowed to fall. Approximately 150 to 200 grams out of the total charge of 600 grams was consumed before the desired conditions of operation were fixed.

Thus the recoveries are low for pressure reactions, and the error is rather considerable with low temperatures and high pressures. These effects should be thoroughly understood in considering the yields obtained. For comparative purposes the results as recorded are of great value and clearly indicate what can be done. At the time of the public demonstration at Columbia University, those present were shown clearly the possibility of making gasoline without evolving gas, once the pressure had been built up. The high concentration of end-product gases in the reaction zone is sufficient to prevent further formation of these undesirable products. With regard to the modern dynamic conception of chemistry, it may be said that the evolved gas enters into chemical union as fast as it is formed; that is, a state of equilibrium exists.

The percentages of recovered oil on the basis of the original oils are given in Table 10, and the average percentages are assembled in Table 11.

TABLE 10.—*Results of cracking various original oils, showing percentage, by weight, of oil recovered.*

Furnace conditions.		Percentage, by weight, recovered from original oils from—				Average percentage recovered.
Temperature.	Pressure.	Pennsylvania.	Oklahoma.	California.	Mexico.	
°C.	*Atmospheres.*					
850	1	9.7	....	....	....	9.7
850	(a)	....	23.1	34.2	....	28.6
800	(a)	....	29.3	43.7	....	36.5
750	12	15.9	20.2	21.1	....	19.1
750	6	16.9	22.2	20.9	....	20.0
750	1	16.5	17.5	20.9	....	18.3
750	(a)	33.9	51.7	47.4	37.3	42.6
650	(a)	....	88.7	....	....	88.7
600	24	37.3	33.9	31.6	....	34.2
600	18	40.5	35.6	33.7	....	36.6
600	12	43.6	62.2	52.5	47.2	51.4
600	6	31.4	37.3	43.9	....	87.5
600	1	59.9	50.8	46.6	40.2	49.4
600	(a)	97.4	100.0	81.8	66.0	85.0
550	24	....	38.1	40.6	....	39.3
550	18	....	55.0	....	....	55.0
500	24	72.5	63.8	53.6	....	63.3
500	18	79.4	....	....	....	79.4
500	12	84.2	88.2	64.8	54.5	72.9
500	6	57.8	91.8	71.5	....	73.7
500	1	94.5	99.0	91.3	72.8	89.4
500	(a)	100.0	99.8	73.6	....	91.1
450	18	....	76.9	....	73.5	75.2
450	12	93.1	87.7	75.4	....	85.4
400	10	....	....	....	....	....

a Vacuum.

Table 11.—*Summary of results of cracking four oils, showing variations in oil recovered under various conditions of temperature and pressure.*

Pressure.	Percentage of oil recovered at temperature of—							
	450° C.	500° C.	550° C.	600° C.	650° C.	750° C.	800° C.	850° C.
Atmospheres.								
(a)	....	91.1	....	85.0	88.7	42.6	36.5	28.6
1	....	89.4	....	49.4	....	18.3	....	9.7
6	....	73.7	....	37.5	....	20.0	....	....
12	85.4	72.9	....	51.4	....	19.1	....	....
18	75.2	79.4	55.0	36.6	....	....	....	....
24	....	63.3	39.3	34.2	....	....	....	....

[a] Vacuum.

Differences in the original oils, whether of a physical or chemical nature, seem to have had little influence on the recoveries. It is shown clearly and is brought out by figure 11 that the percentage of recovery decreases with increase of temperature. There seems to have been no characteristic variation among the original oils, for the maximum

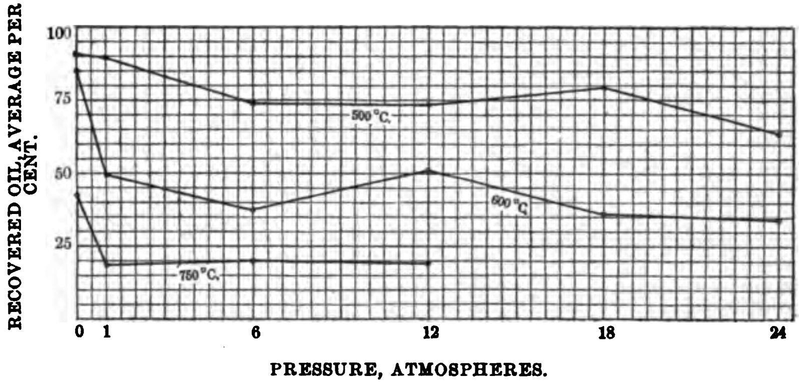


Figure 11.—Curves representing average results of cracking four oils, showing influence of varying temperatures and pressures on quantity of oil recovered on basis of original oil. Results for higher pressures low on account of oil consumed in building up pressures.

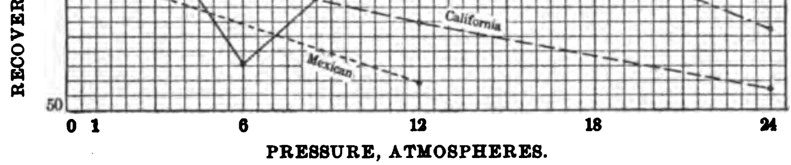


Figure 12.—Curves showing percentages of oil recovered from cracking four oils under varying pressures at 500° C. Results for higher pressures low on account of oil consumed in building up pressures.

as well at the minimum, recovery for these oils at the varying temperatures and pressures recorded seems to have been uniform.

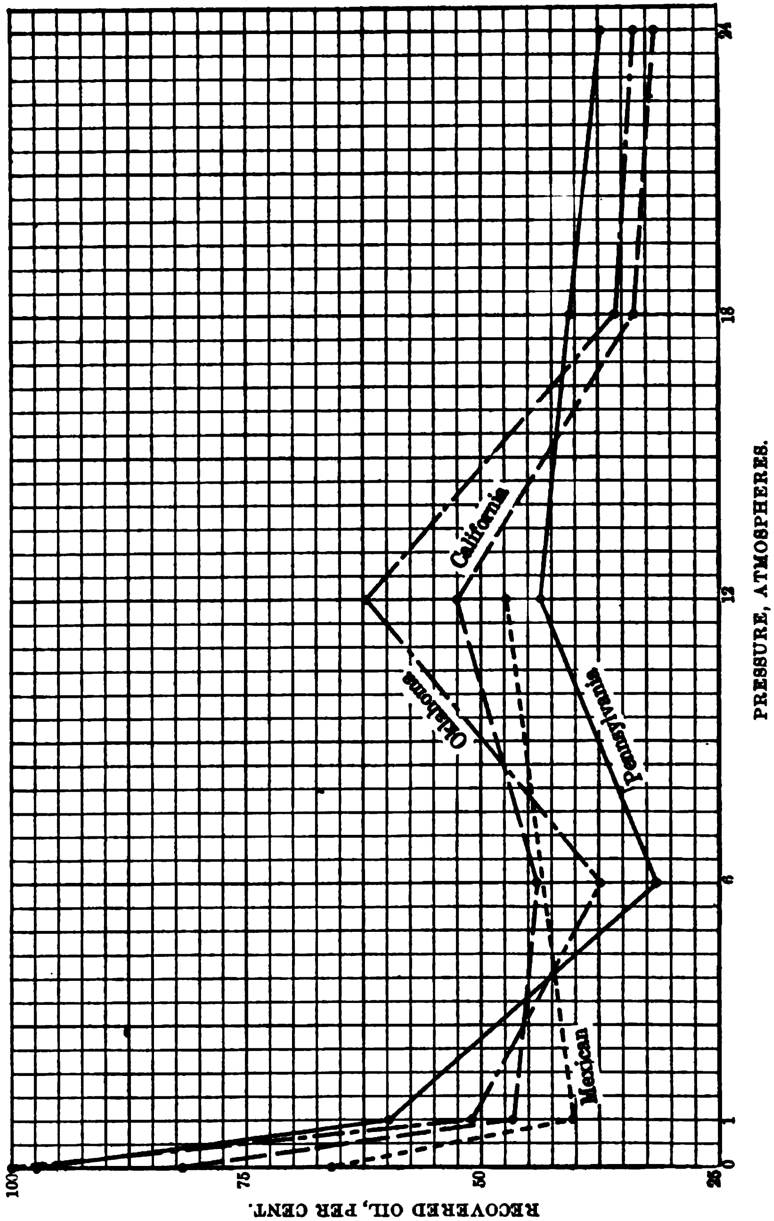


FIGURE 13.—Curves showing percentages of oil recovered from cracking four oils under varying pressures at 600° C. Results for higher pressures low on account of oil consumed in building up pressures.

At the higher temperatures there appears to have been a tendency toward increased yields at a pressure of 12 atmospheres, and a falling off for the higher pressures at the same temperatures. In considering yields under increased pressure, however, the conditions of the experiments must not be overlooked. These of necessity were distinctly disadvantageous to pressure work. The influence of pressure at constant temperatures on quantitative yields is graphically shown in figures 12, 13, and 14.

CARBON FORMATION.

The percentage of carbon formed in any cracking process is likewise a factor of the greatest industrial importance, for as has been pointed out, the formation of carbon in tubes of small internal diameter has been considered the greatest objection to their use in cracking processes.

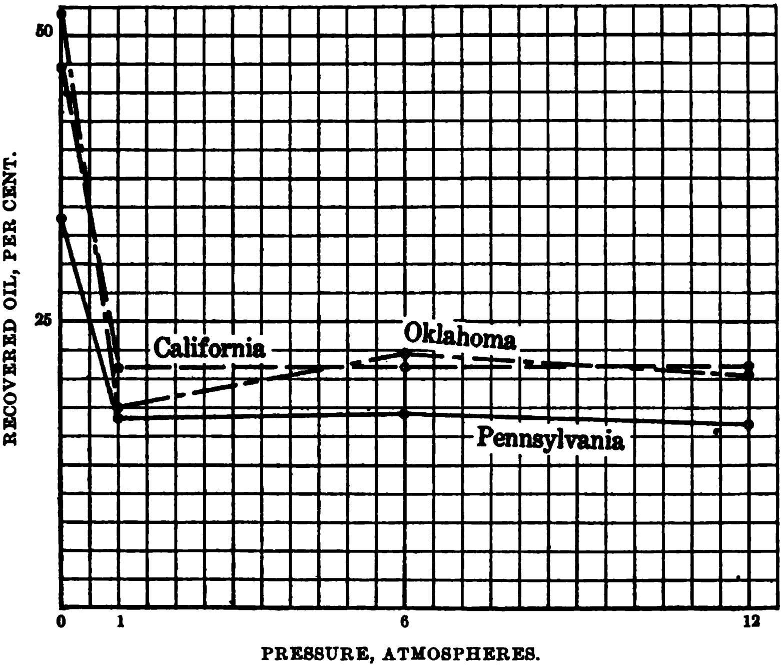


FIGURE 14.—Curves showing percentages of oil recovered from cracking three oils under varying pressures at 750° C. Results for higher pressures low on account of oil consumed in building up pressures.

Without efficient means of removing carbon that might form in considerable quantities, the operation of any process would not be continuous. The rapidity with which carbon forms and the cost of removing carbon will be decisive factors in determining the commercial practicability of a process.

With the machine used in the present experiments little carbon is formed in the range of temperature and pressure favorable for gasoline formation. A much greater quantity of carbon is formed at the temperatures and pressures favorable for the formation of aromatic hydrocarbons. Happily, effective mechanical means for removing this carbon have been developed in the large-scale commercial operation, so that in equipment for production on a commercial scale carbon deposition is not a serious factor. The percentage of carbon formed in the experiments was determined by scraping the interior of the tubes after each run or experiment and weighing the solid matter thus removed. The values thus obtained for the various oils used are given in table 12, following.

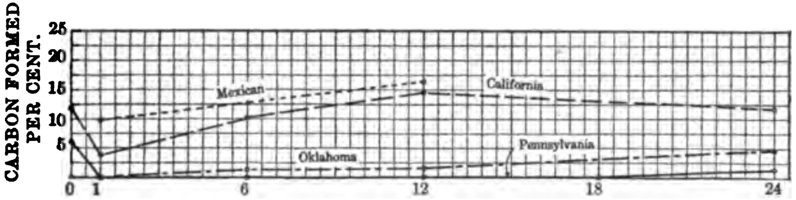
TABLE 12.—*Results of cracking four oils under various conditions of temperature and pressure showing percentage of carbon formed.*

Furnace conditions.		Percentage of carbon formed from original oil from—				Average proportion recovered.
Temperature.	Pressure.	Pennsylvania.	Oklahoma.	California.	Mexico.	
°C.	*Atmospheres.*					
850	1	7.4	....	....	....	7.4
850	(a)	....	5.8	7.1	....	6.4
800	(a)	....	4.1	6.9	....	5.5
750	12	24.3	24.6	38.8	....	29.2
750	6	15.3	20.0	36.2	....	23.8
750	1	3.7	11.3	9.9	....	8.3
750	(a)	16.1	4.1	8.7	10.5	13.1
650	(a)	....	2.2	....	....	2.2
600	24	7.5	17.2	26.7	....	17.1
600	18	5.3	13.2	27.5	....	15.3
600	12	3.6	6.7	19.3	13.1	10.7
600	6	2.4	9.6	16.2	....	9.4
600	1	0.6	3.1	12.3	16.4	8.1
600	(a)	0.0	....	13.6	9.0	7.5
550	24	....	9.2	21.1	....	15.1
550	18	....	7.0	....	....	7.0
500	24	1.2	4.6	11.7	....	5.8
500	18	0.0	....	....	....	0.0
500	12	0.0	1.4	14.5	16.2	8.0
500	6	0.0	1.2	10.1	....	3.8
500	1	0.0	0.0	3.7	9.8	3.6
500	(a)	6.0	0.0	11.9	....	5.9
450	18	....	0.0	....	....	0.0
450	12	5.1	0.0	2.3	....	2.5
400	10	....	....	....	13.8	13.8

[a] Vacuum.

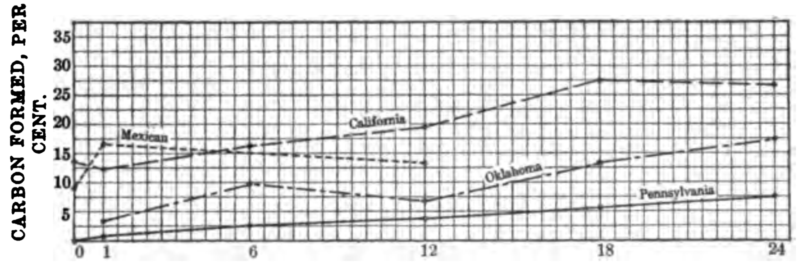
The amount of carbon formed, together with the volume of evolved fixed gases, is a valuable index of the percentage of total decomposition as contrasted with a partial alteration or cracking.

The differences among percentages of carbon formation may be attributed to variations in both the physical and the chemical properties of the oils studied. In the first place, the average size of the hydrocarbon molecules differed widely, the heavier oils containing a larger percentage of high-boiling constituents which were decomposed more rapidly by heat and formed higher percentages of carbon and fixed gases. The asphaltic-base California and Mexican oils show a consistently higher carbon percentage at the varying temperatures than the paraffin-base Pennsylvania oil. The mixed-base Oklahoma oil is intermediate. The Pennsylvania oil, which consistently deposited the least carbon, was a water-white burning oil (kerosene), 90 per cent of which distilled over at a temperature of less than 250° C. The Oklahoma fuel oil gave a distillation cut of 22 per cent below 300° C., and the California oil gave a cut of only 17 per cent below 300° C., whereas the Mexican crude, which was "lighter" than the California oil, gave a distillation cut of 37 per cent below 300° C. This Mexican oil seemed to have the peculiar physical property of causing considerable heavy tarry material to be included in the carbon deposit, so that the figures for carbon formation are high, and the figures for yields of recovered oil and the specific gravities of these oils are correspondingly low. However, experience with the larger tubes has served to indicate that on a larger scale much more favorable results can be expected with this oil than were obtained with the small 1½-inch tubes of the experimental apparatus. The characteristic difference is shown by figures 15, 16, and 17.



PRESSURE, ATMOSPHERES.

FIGURE 15.—Curves showing percentages of carbon formed from cracking four oils under varying pressures at 500° C.



PRESSURE, ATMOSPHERES.

FIGURE 16.—Curves showing percentages of carbon formed from cracking four oils under varying pressures at 600° C.

The increase in the amount of carbon formation at the temperatures favorable to the manufacture of aromatic hydrocarbons as compared with the relatively lower percentages of formation under the conditions favorable for gasoline formation, is probably to be explained on the theory that production of gasoline by cracking is a simple dissociation process. In the production of aromatic hydrocarbons it is necessary in a considerable degree both to break down the larger molecules to ethylene or acetylene hydrocarbons and then to polymerize these unsaturated hydrocarbons. Experiment has proven that aromatic hydrocarbons are polymerization products of acetylene and ethylene hydrocarbons as well as the results of the splitting of polycyclic compounds.[a] It thus becomes necessary to employ the high temperatures favorable for olefin and acetylene formation and the high pressures favorable for polymerization. The combination of these two factors results in a higher percentage of carbon formation.

Thus, it is evident that because of the increase in carbon formation with increase in temperature, it is not commercially desirable to employ the highest temperatures favorable for forming the desired reaction products, but instead to employ a mean temperature that will give a favorable degree of conversion with a reasonable production of carbon. This consideration is of moment only in the manufacture of benzene and toluene.

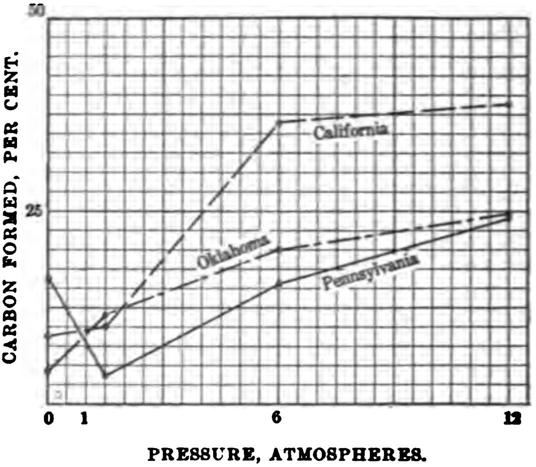


FIGURE 17.—Curves showing percentages of carbon formed from cracking three oils under varying pressures at 750° C.

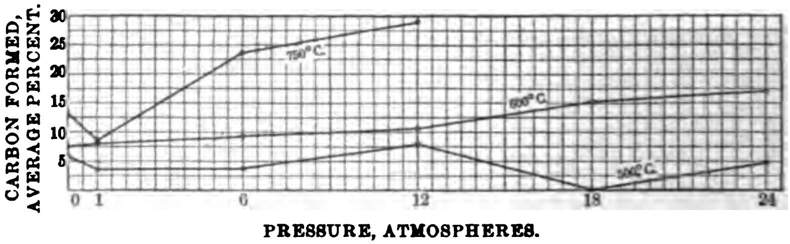


FIGURE 18.—Curves representing average results of cracking four oils, showing variations in carbon formation with variations in temperature and pressure.

The average percentages of carbon formation at the varying temperatures and pressures are presented in Table 13, following. The curves of average percentage formation at temperatures of 500°, 600°, and 750° C., are shown in figure 18.

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[a] Ipatiew, W., Catalytic reactions at high temperatures and pressures; Polymerization of ethylene; hydrocarbon at high temperatures and pressures: Ber. Deut. chem. Gesell., Jahrg. 44, B. 3, 1911, p. 2978, Meyer, Richard, Pyrogenic Acetylen-Kondensationen: Ber. Deut. chem. Gesell., Jahrg. 45, Bd. 2; 1912. pp. 1609–1633; Meyer, Richard, and Tanzen, A., Pyrogene Acetylen-Kondensationen: Ber. Deut. chem. Gesell., Jahrg. 46, Bd. 3, 1913, pp. 3183–3199.

TABLE 13.—*Summary of results of cracking the four oils, showing average amount of carbon formed and variations in amount under various conditions of temperature and pressure.*

Pressure.	Percentage of carbon recovered at temperature of—							
	450° C.	500° C.	550° C.	600° C.	650° C.	750° C.	800° C.	850° C.
Atmospheres.								
(a)	....	5.9	....	7.5	2.2	13.1	5.5	6.4
1	....	3.6	....	8.1	....	8.3	....	7.4
6	....	3.8	....	9.4	....	23.8	....	....
12	2.5	8.0	....	10.7	....	29.2	....	....
18	0.0	0.0	7.0	15.3	....	....	....	....
24	....	5.8	15.1	17.1	....	....	....	....

[a] Vacuum.

An average of irregular sets of figures, such as those obtained for these oils, which does not take into account the basic differences in the oils used, is necessarily inaccurate. However, such an average shows the character of the results to be expected at the various reaction temperatures, and brings out clearly the characteristic increase of carbon formation with increase in temperatures. In addition, it indicates that carbon formation, as is to be expected, is favored not alone by increase in temperature but also by increase in pressure for a given temperature.

SPECIFIC GRAVITIES OF RECOVERED OILS.

The results of determinations of the specific gravities of the recovered oils are presented in Table 14. The averages of the values for these different oils at each temperature and pressure are given in Table 15. The average values tend to give a clearer conception of the changes occurring with increasing temperatures and pressures, although on account of the conditions prevailing in the runs they are not strictly accurate.

TABLE 14.—*Results of cracking the four oils under various conditions of temperature and pressure showing specific gravities of recovered oils.*

Furnace conditions.		Specific gravity of recovered oil from original oil from—				Average specific gravity.
Temperature.	Pressure.	Pennsylvania.	Oklahoma.	California.	Mexico.	
° C.	*Atmospheres.*					
850	1	1.026	....	....	....	1.026
850	(a)	....	.997	1.025	....	1.011
800	(a)	....	.963	1.017	....	.990
750	12	.910	.965	1.022	....	.965
750	6	1.020	1.040	1.025	....	1.028
750	1	1.012	1.039	1.092	....	1.048
750	(a)	.905	.925	.963	.992	.946
650	(a)	....	....	....	....	....
600	24	.962	1.008	1.030	....	1.000
600	18	.926	.973	.997	....	.965
600	12	.918	.907	.996	.941	.940
600	6	.885	.931	.984	....	.933
600	1	.840	.906	.966	.992	.926
600	(a)	.811	.899	.960	.942	.903
550	24	....	....	.991	....	.991
550	18	....	.908	....	....	.908
500	24	.826	.877	.942	....	.882
500	18	.819	....	....	....	.819
500	12	.808	.870	.925	.918	.880
500	6	.810	.867	.928	....	.868
500	1	.799	.872	.939	.930	.885
500	(a)	.802	.901	.950	....	.884
450	18	....	.882	....	....	.882
450	12	.809	.833	.924	....	.868
400	10	....	....	....	.934	.934
Specific gravity of original oil.		.802	.894	.970	.986	....

[a] Vacuum.

TABLE 15.—*Summary of results of cracking four oils under various conditions of temperature and pressure, showing average specific gravities of recovered oils.*

Pressure.	Specific gravity of cracked oils at temperature of—							
	450° C.	500° C.	550° C.	600° C.	650° C.	750° C.	800° C.	850° C.
Atmospheres.								
(a)	....	0.884	....	0.903	....	0.946	0.990	1.011
1	....	.885	....	.926	....	1.048	....	1.026
6	....	.868	....	.933	....	1.028	....	....
12	0.868	.880	....	.940	....	.965	....	....
18	.882	.819	0.908	.965	....	....	....	....
24	....	.882	.991	1.000	....	....	....	....

[a] Vacuum.

DISCUSSION OF TABULATED RESULTS.

For the purpose of more clearly depicting the effect of subjecting the oils to the experimental temperatures and pressures, the curves in figures 19, 20, and 21 have been prepared, showing the specific gravities of the recovered oils obtained from each series of experiments.

It will be noted that the differences in specific gravity among the recovered oils are, in general, in the same order as the gravity differences among the original oils. The reaction products of the Pennsylvania water-white distillate are the lightest, those from the Oklahoma residuum rank next, and those from the California crude are the heaviest. The specific gravities of the recovered oils from the Mexican crude are less than those from the California crude although the original California oil was the lighter. This difference is to be explained by the tendency of heavy constituents in the Mexican oil to become mechanically attached to the carbon.

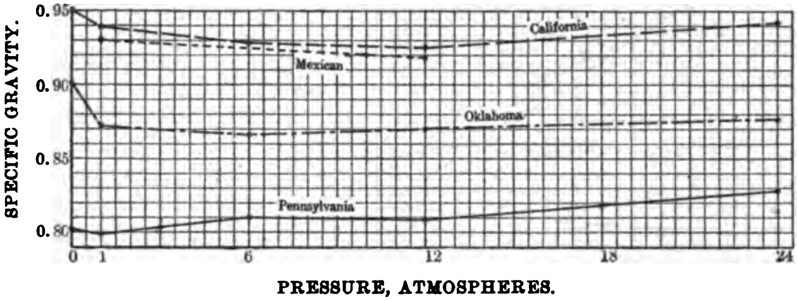

FIGURE 19.—Curves showing specific gravities of oils recovered from cracking oils under varying pressures at 500° C.

In general the results clearly indicate that increase in the specific gravities of recovered oils is coincident with increase in temperature (see figs. 19, 20, and 21). There is also a marked tendency toward increase in gravity with increase of pressure at any given temperature. Deviations from this general rule are to be explained on the basis of experimental error.

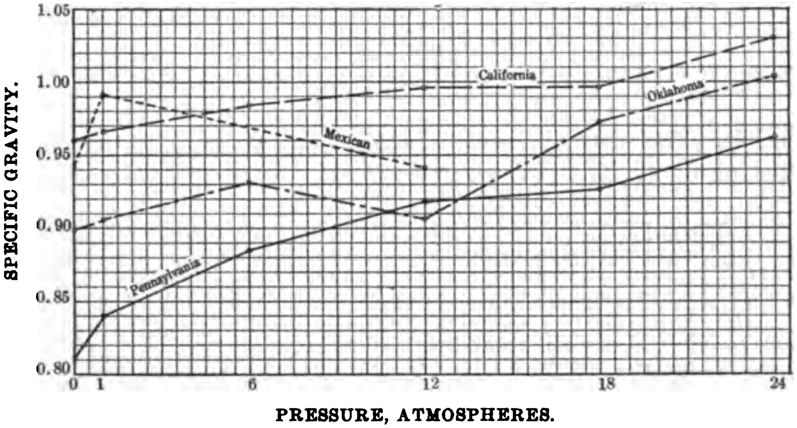

FIGURE 20.—Curves showing specific gravities of oils recovered from cracking oils under varying pressures at 600° C.

The average specific gravities of the recovered oils have been plotted in figure 22. Although this manner of presentation is unscientific, in view of the basic differences in the original oils, it gives a good general idea of the relative influences of temperature and pressure on the oils that may be used in this type of cracking process.

It has been found that volatility and specific gravity are the most valuable physical constants for use in determining the nature of a given oil. The specific-gravity method of determining yields of desired products in the recovered oils which has given satisfactory results has been developed through experimental research extending over a period of years. The application of the method is more fully

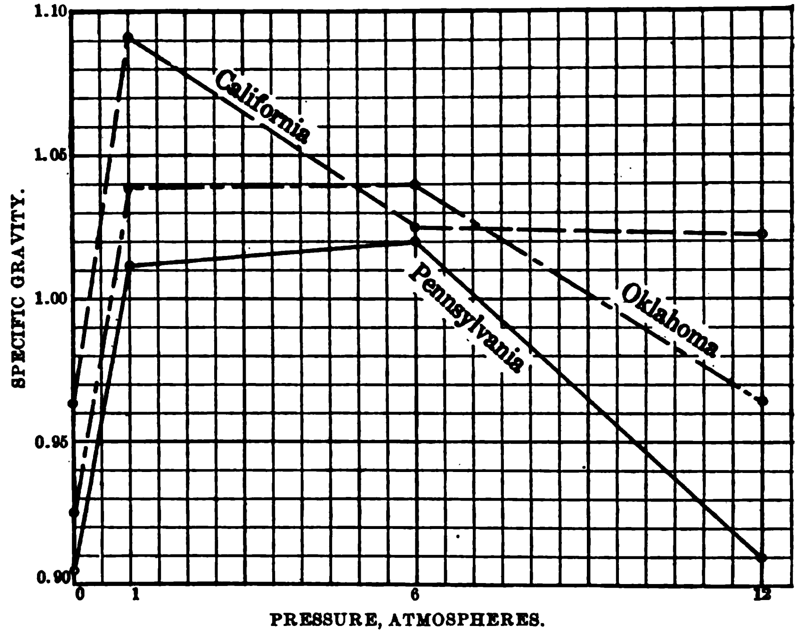

FIGURE 21.—Curves showing specific gravities of oils recovered from cracking oils under varying pressures at 750° C.

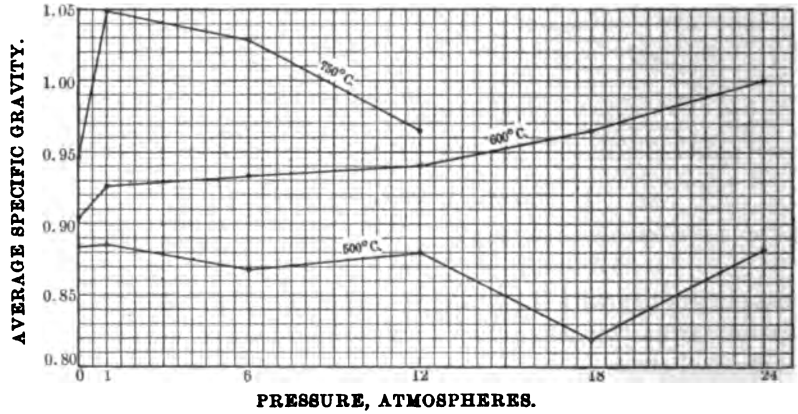

FIGURE 22.—Curves representing average results of cracking oils, and influence of varying temperatures and pressures on specific gravities of oils recovered.

discussed subsequently. The method has been checked with nitration methods of analysis, and has been found dependable. It is as reliable as any analytical method, and is particularly useful in the quantitative determination of the aromatic hydrocarbons in a given oil. The values assumed for calculating yields have been experimentally checked and proved to be accurate. The chief recommendations of the method are the greater facility of making determinations and its accuracy.

VISCOSITY OF OIL RECOVERED.

In a number of the experiments the quantity of oil recovered was so small that viscosity measurements could not be made.

The values that were obtained are given in Table 16. The most noticeable feature in connection with these figures is the fact that, although the original oils differ widely in viscosity, the figures for a given temperature and pressure show only slight variations. This is one of the facts that tend to establish the conclusion that the conditions under which an oil is treated are of much greater influence than the physical and chemical properties of the oil. For example, the ratio of viscosity between the Pennsylvania water-white distillate and the Kern River crude was approximately 1 to 35, whereas the average viscosity ratio for the recovered oils from the two sources was less than 1 to 3. At pressures above 1 atmosphere this ratio was approximately 1 to 1.5.

The table shows that for temperatures up to 750° C. the viscosity decreases with temperature and pressure. Experimental evidence obtained at higher temperatures proves that there is a progressive increase in viscosity at temperatures of 800° C. and above. Table 16 follows:

TABLE 16.—*Results of cracking five oils, showing viscosities of oils recovered.*

Furnace conditions.		Viscosity of recovered oil from original oil from—				
Temperature.	Pressure.	Pennsylvania.	Oklahoma.	California.	Mexico.	Russia.
°C.	*Atmospheres.*					
800	[a]	....	1.64	3.95	....	....
750	[a]	1.04	1.44	....	2.70	....
600	12	1.11	1.48	1.79	....	....
600	6	1.00	1.08	1.15	....	....
600	1	....	1.27	1.98	1.96	....
600	[a]	1.04	7.29	....	6.73	....
550	24	....	1.05	....	....	....
500	24	.97	1.25	1.35	....	....
500	18	.96	....	....	....	....
500	12	.98	1.32	1.35	1.15	1.44
500	6	1.00	1.54	1.52	....	....
500	1	1.00	2.51	6.16	1.88	....
500	[a]	1.04	13.42	....	....	....
450	18	....	1.50	2.71	....	....
450	12	1.02	1.78	....	4.43	....
400	10	....	....	....	....	....
Vicosity of original oil..........		1.05	35.40	35.70	Over 350	11.92

[a] Vacuum.

It has not been considered necessary to average the values for the various oils, as consideration of the results obtained for any one oil will be found sufficiently suggestive. For ease of comparison, the viscosity values for the Oklahoma oil, for which the most complete data were obtained, are grouped in Table 17. The table shows clearly that for runs up to 750° C. there is a decrease in viscosity with increase of temperature and pressure. The single figure obtained for a temperature of 800° C. establishes that this relation will not hold true for higher temperatures, as is to be expected. The like figure for the California oil is in substantial accord. Table 17 follows:

TABLE 17.—*Results of cracking Oklahoma fuel-oil residuum, showing effects of varying temperatures and pressures on viscosities of recovered oils.*

Pressure.	Viscosity of oils recovered at temperature of—					
	450° C.	500° C.	550° C.	600° C.	750° C.	800° C.
Atmospheres.						
(a)		13.42		7.29	1.44	1.64
1		2.51		1.27		
6		1.54		1.08		
12	1.73	1.32		1.48		
18	1.50					
24		1.25	1.05			

[a] Vacuum.

GAS FORMATION.

The volume of fixed and permanent gases (CH_4, H_2, C_2H_6, etc.) is of large commercial importance in any cracking process. These gases are undesirable, and too great a yield means a considerable increase in the cost of manufacturing the liquid reaction products.

In the conventional cracking processes, that treat a large volume of liquid at a given time in a still or other large retort, the evolution of fixed gases in the continued decomposition of the hydrocarbon molecules constitutes a serious drawback to commercial efficiency. The gases, being lighter than the liquids, are continually passing up and out of the sphere of reaction.

This volume loss due to evolved gases is not, however, the most serious objection. If gasoline formation is the end in view, each cubic foot of gas emitted means a proportional increase in the quantity of unsaturated liquid hydrocarbons that remain.

A little methane would not be serious in itself, but, with its high hydrogen content, it would serve to saturate much of the remaining gasoline hydrocarbons. This deficiency in saturation makes a considerable difference in the refining loss on washing with sulphuric acid. Experiments showed losses from washing with sulphuric acid varying from 2 to 50 per cent. The sulphuric-acid loss is entirely a function of the unsaturated hydrocarbons remaining in the oil; the degree of unsaturation is in turn a function of the temperature and pressure prevailing at the time of cracking. For a given temperature, the percentage of unsaturated hydrocarbons appears to decrease indefinitely with increase in pressure. For a given pressure the degree of unsaturation increases with temperature.

Gas formation is favored by high temperature and, therefore, must occur in considerable degree when hydrocarbons are cracked under a high temperature. In the benzene-toluene process a relatively large amount of gas is evolved, this being necessarily one of the accompanying features of the complete decomposition of the oil necessary to bring about the formation of aromatic hydrocarbons.

An important question is that concerning the commercial value of gas so formed. In some experiments, conditions did not permit the obtaining of gas samples and in others the sampling was faulty. However, the data obtained are sufficient to bring out several important points.

Actual tank readings of volumes have been transformed so that the figures given represent the volumes (both in liters and in cubic feet) of gases generated by 1 kilogram of oil. These volumes represent air-free gas (hydrogen and hydrocarbons) and are calculated for standard conditions of 0° C. temperature and 760 mm. pressure.

The gas produced by the benzene-toluene process represents a valuable by-product that will help to offset the loss caused by the decomposition of the oil into fixed gases and carbon. In fact, it may even develop later that the permanent gases will be the primary products and the condensable liquids the by-products.

The most important commercial features of a gas are, first, the percentage of olefin hydrocarbons (illuminants) it contains, and, second, its heating value. Figures are presented in Table 18 that indicate the properties of the gases evolved in the course of the present experiments.

The results obtained for the various oils agree in a general way as to the volume of gas produced and clearly indicate that, as with the other factors, conditions of operation are of more importance than the chemical and physical differences of the original oils. Only a small volume of the gas is evolved at the temperature favorable for gasoline production. The gas produced in the benzene-toluene process is sufficient, however, to provide adequate heat for the cracking operations and in addition to leave a large surplus available for general plant purposes or for sale.

A fair agreement is found among the four original oils in the matter of illuminant production. The results again emphasize the predominating influence of temperature, pressure, and concentration, as compared with the nature of the oil.

The calorific values of the gases, calculated on the basis of British thermal units per cubic foot of gas, prove that the gases produced in the benzene-toluene process constitute a most satisfactory fuel. The heating values are greater than those of good natural gas, owing to the presence of considerable proportions of ethane and ethylene.

The experiments being concerned more with the nature and the quantity of the liquid reaction products than with the gaseous products, attention was concentrated on the former. Too much reliance should not be placed on the percentages of gas formed. As previously stated, in the authors' small experimental operations building up pressures consumed a large quantity of the initial charge of any oil treated. In consequence the percentages of gas formed were greater for all pressure reactions than they would be in large-scale operations. The results are of value nevertheless for comparison and show in a general way what may be expected under various temperature and pressure conditions. The results mentioned are shown in Table 18. The percentages of gas formed are shown in figures 23, 24, and 25.

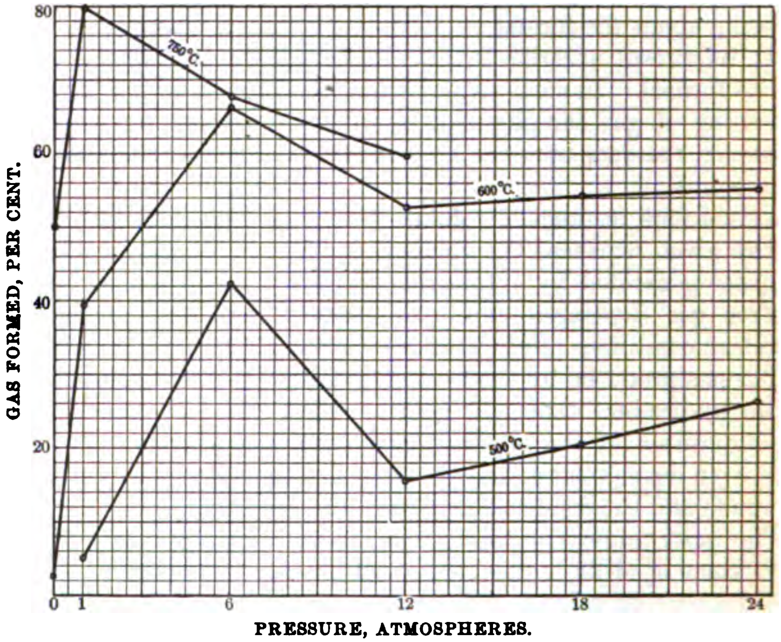

FIGURE 23.—Curves showing percentages of gas formed from cracking a Pennsylvania refined burning oil with varying temperatures and pressures. Results for higher pressures are high because of oil consumed in building up pressures.

Table 18.—*Results of cracking five oils under various conditions of temperature and pressure, showing quantities of gas formed.*

PENNSYLVANIA WATER-WHITE BURNING OIL.

Furnace conditions.		Gas formed.		Furnace conditions.		Gas formed.	
Temperature, °C.	Pressure, atmospheres.	Percentage by weight.[a]	Cubic feet per kilogram of oil used.[b]	Temperature, °C.	Pressure, atmospheres.	Percentage by weight.[a]	Cubic feet per kilogram of oil used.[b]
850	1	82.9	28.9	600	1	39.5	....
750	12	59.8	....	600	(c)	2.6	0.59
750	6	67.8	21.9	500	24	26.3	....
750	1	79.8	21.6	500	18	20.3	....
750	(c)	50.0	....	500	12	15.8	....
600	24	55.2	....	500	6	42.2	1.9
600	18	54.2	....	500	1	5.5	....
600	12	52.8	8.48	500	(c)	....	.187
600	6	66.2	....	450	12	1.8	....
OKLAHOMA FUEL-OIL RESIDUUM.							
850	(c)	71.1	20.1	600	(c)	0.0	1.35
800	(c)	66.6	....	550	24	52.7	11.1
750	12	55.2	....	550	18	38.0	....
750	6	57.8	....	500	24	31.6	....
750	1	71.2	21.1	500	12	10.4	....
750	(c)	44.2	10.6	500	6	7.0	1.89
600	24	48.9	....	500	1	1.0	3.11
600	18	51.2	13.9	500	(c)	.2	....
600	12	31.1	6.65	450	18	23.1	....
600	6	53.1	12.6	450	12	12.3	....
600	1	46.1	10.2				
CALIFORNIA (KERN RIVER) CRUDE OIL.							
850	(c)	58.7	15.15	600	1	41.1	7.23
800	(c)	49.4	....	600	(c)	4.6	1.17
750	12	40.1	....	550	24	38.3	9.72
750	6	42.9	19.65	500	24	34.7	....
750	1	69.2	17.50	500	12	20.7	....
750	(c)	43.9	3.70	500	6	18.4	3.64
600	24	41.7	....	500	1	5.0	12.1
600	18	38.8	13.60	500	(c)	14.5	2.66
600	12	28.2	8.75	450	12	22.3	....
600	6	39.9	11.10				
MEXICAN CRUDE OIL.							
750	(c)	52.2	11.3	500	12	29.3	5.58
600	12	39.7	8.82	500	1	17.4	6.22
600	1	43.4	9.12	400	10 to 11	12.7	....
600	(c)	25.0	....				
RUSSIAN RESIDUUM OIL.							
675	12	50.0	17.8	500	12	22.7	....

[a] Percentage of gas formed was calculated by difference. Figures are necessarily high, as experimental losses are included.

[b] Sum of the quantities of illuminants, methane, ethane, and hydrogen as indicated by analyses of gas samples.

[c] Vacuum.

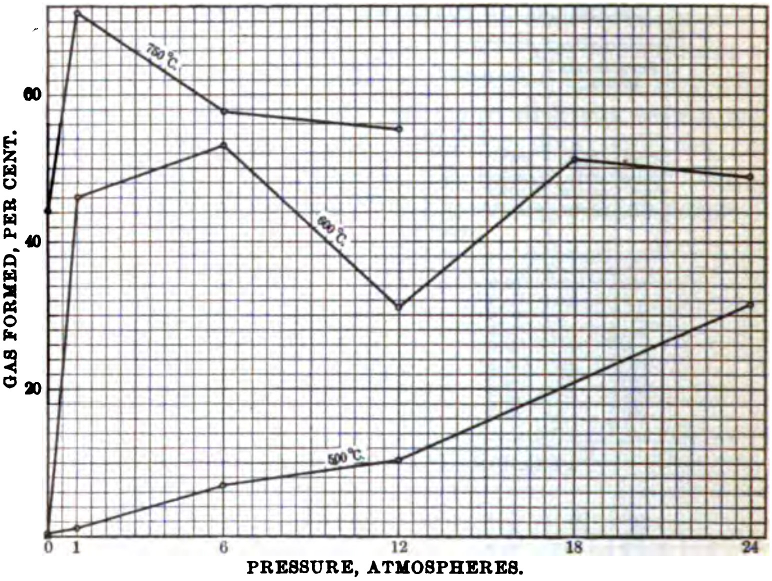

Figure 24.—Curves showing percentages of gas formed from cracking Oklahoma fuel-oil residuum with varying temperatures and pressures. Results for higher pressures are high, owing to oil consumed in building up pressures.

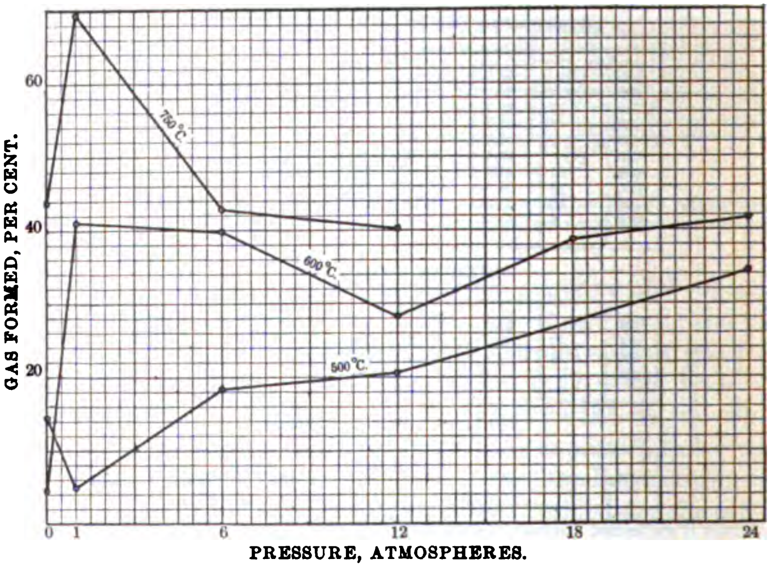

Figure 25.—Curves showing percentages of gas formed from cracking a California (Kern River) crude oil with varying temperatures and pressures. Results for higher pressures are high because of oil consumed in building up pressures.

GASOLINE FORMATION.

To set a satisfactory standard for what constitutes gasoline is difficult. The upper limit of temperature of the so-called gasoline fraction in the distillation of refinable crude petroleum is being extended from 150° to 170° C., and perhaps higher. Furthermore, there is a wide variance in the physical properties and chemical constitution of the oils that are commercially marketed under the name of gasoline. These differences are not of primary importance so far as this report is concerned, but they are mentioned to show the difficulty of adopting a reasonably satisfactory definition. For present purposes the magnitude of the distillation cut at 150° C. obtained from recovered oil is used as a measure of its gasoline content. A report now in preparation will deal with the characteristics of the various commercial gasolines and their suitability for various uses.

The calculations on which the figures for quantitative yields are based were made as follows:

The amount of the distillation cut below 150° C. for the original oil was deducted from the like figure for the recovered oil. The figure for the recovered oil was then reduced to terms of the original oil, showing the degree of transformation into gasoline hydrocarbons boiling below 150° C. Tables 19 and 20 show the results obtained with the four oils mentioned.

TABLE 19.—*Results of cracking the four oils under various conditions of temperature and pressure, showing percentage of gasoline (distillation cut up to 150° C.) obtained, calculated on basis of original oil.*[a]

Furnace conditions.		Percentage of gasoline formed by cracking oil from—				Average amount of gasoline formed.
Temperature.	Pressure.	Pennsylvania.	Oklahoma.	California.	Mexico.	
° C.	*Atmospheres.*					
850	1	2.3	....	....	....	2.3
850	(b)	....	4.2	4.8	....	4.5
800	(b)	....	5.6	8.8	....	7.2
750	12	5.5	8.8	10.0	....	8.1
750	6	6.2	5.8	8.2	....	6.7
750	1	4.3	4.7	5.1	....	4.7
750	(b)	4.5	7.5	4.0	4.6	5.1
650	(b)	....	3.0	....	....	3.0
600	24	13.3	12.9	10.7	....	12.3
600	18	15.8	16.6	13.0	....	15.1
600	12	16.3	12.1	11.8	11.6	12.9
600	6	10.0	14.1	15.7	....	13.3
600	1	13.4	12.7	10.6	8.1	11.2
600	(b)	.0	2.5	5.3	3.1	3.6
550	24	....	12.4	12.1	....	12.3
550	18	....	17.3	....	....	17.3
500	24	18.1	14.0	13.5	....	15.2
500	18	15.3	13.8	....	....	14.6
500	12	16.0	10.9	14.3	14.6	13.7
500	6	5.9	4.7	13.7	10.6	8.7
500	1	2.7	.0	7.0	....	4.9
500	(b)	.0	....	1.1	....	1.1
450	18	....	9.4	....	....	9.4
450	12	6.5	8.8	7.2	....	7.5
400	10	....	....	....	7.1	7.1

[a] Results for higher pressures low on account of oil consumed in building up pressures.
[b] Vacuum.

TABLE 20.—*Results of cracking four oils, showing effects of temperature and pressure on average percentage of gasoline formed.*[a]

Pressure.	Percentage of gasoline formed at temperature of—							
	450° C.	500° C.	550° C.	600° C.	650° C.	750° C.	800° C.	850° C.
Atmospheres.								
(b)	....	1.1	....	3.6	3.0	5.1	7.2	4.5
1	....	4.9	....	11.2	....	4.7	....	2.3
6	....	8.7	....	13.3	....	6.7	....	....
12	7.5	13.7	....	12.9	....	8.1	....	....
18	9.4	14.6	17.3	15.1	....	....	....	....
24	....	15.2	12.3	12.3	....	....	....	....

[a] Results for higher pressures low, owing to oil consumed in building up pressure.
[b] Vacuum.

COMMENTS ON RESULTS OF CRACKING OILS TO OBTAIN GASOLINE.

The ease with which the gasoline process can be operated and its freedom from mechanical difficulties makes the highly favorable results shown by these experiments of fundamental interest to refiners and to that part of the public desiring a cheap and satisfactory motor fuel.

In considering the percentages of gasoline formation presented in Table 19 the reader should remember that the small quantities of oil used for each experiment, and the necessity of consuming a relatively high percentage of the charge to build up pressures, made the yield of gasoline much lower than can be expected in large-scale operations in which such losses would be so minimized as to be almost negligible.

In addition, condensation was by no means as efficient as that which would be obtained in large-scale operations. The results from the use of the larger tubes indicated that a considerable percentage of the lighter and more volatile liquid products were mechanically carried over by the fixed or permanent gases, and hence were lost so far as the recovered oil was concerned.

The most noticeable feature of the results presented is the agreement in the values for the various oils used for the respective temperatures and pressures. Here again it is evident that differences in the original oils are of secondary importance as compared with the effect of conditions of temperature and pressure.

As would be expected, the higher temperatures, for example 700° C., were distinctly unfavorable for gasoline formation. The degree of formation of the end products, carbon and gas, is too great. The results presented in the table indicate that temperatures ranging between 500° and 600° C., preferably in the neighborhood of 550° C., are most favorable for gasoline formation. Further experiments with large-size tubes have confirmed this indication. Figure 26, showing the curves of average percentages of gasoline formation for the oils used, clearly indicates the favorable range of temperature of the process.

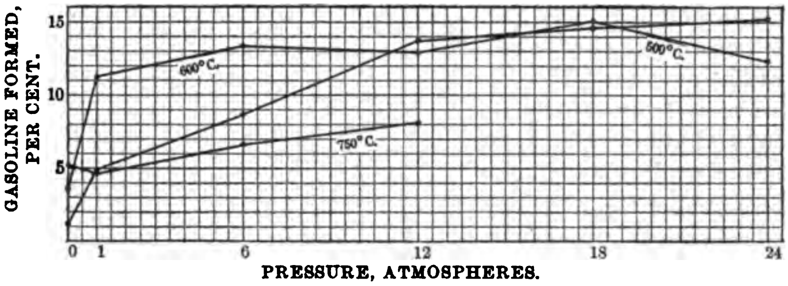

FIGURE 26.—Curves representing average results of cracking oils, showing influence of varying temperatures and pressures on formation of gasoline (hydrocarbons distilling up to 150° C.). Results for higher pressures low, owing to oil consumed in building up pressure.

Figures 27, 28, and 29 give the percentages of gasoline formed from the Pennsylvania, Oklahoma, and California oils at constant temperatures of 500°, 600°, and 750° C., and at varying pressures. That high pressures are favorable for the desired reactions is clearly indicated as the yield increased progressively with pressure. The contrary indications for the pressure of 24 atmospheres are to be explained on the basis of the greater volume losses due to the building up of high pressures.

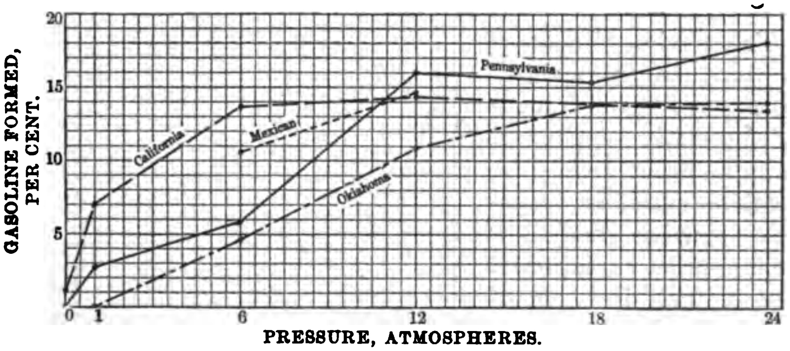

FIGURE 27.—Curves showing percentages of gasoline (hydrocarbons distilling up to 150° C.) formed from cracking four oils under varying pressures at 500° C. Results for higher pressure low, owing to oil consumed in building up pressure.

SPECIFIC GRAVITIES OF THE TWO GENERAL CLASSES OF LOW-BOILING HYDROCARBONS RECOVERED.

It should be noted that although there seems to be no characteristic difference between the percentage results for distillation cuts at 150° C. obtained from runs at 500° and at 600° C., there were considerable differences among the physical and chemical properties of products obtained in the same quantity at the temperatures mentioned. This detail may be observed by comparing the figures for specific gravity. The magnitudes of the differences are indicated in Table 21.

The obvious inference is that the "gasolines" formed at 600° C. contain aromatic compounds, as in no other way is the combination of high specific gravity and low boiling point explainable. The actual isolation of benzene and toluene from these distillates and their identification by chemical reactions was brought about to furnish absolute assurance that their formation had been accomplished.

As far as the general features of the gasoline are concerned, it possesses the characteristic odor of cracked reaction products, but may be freed readily from the greater part of this odor by treatment

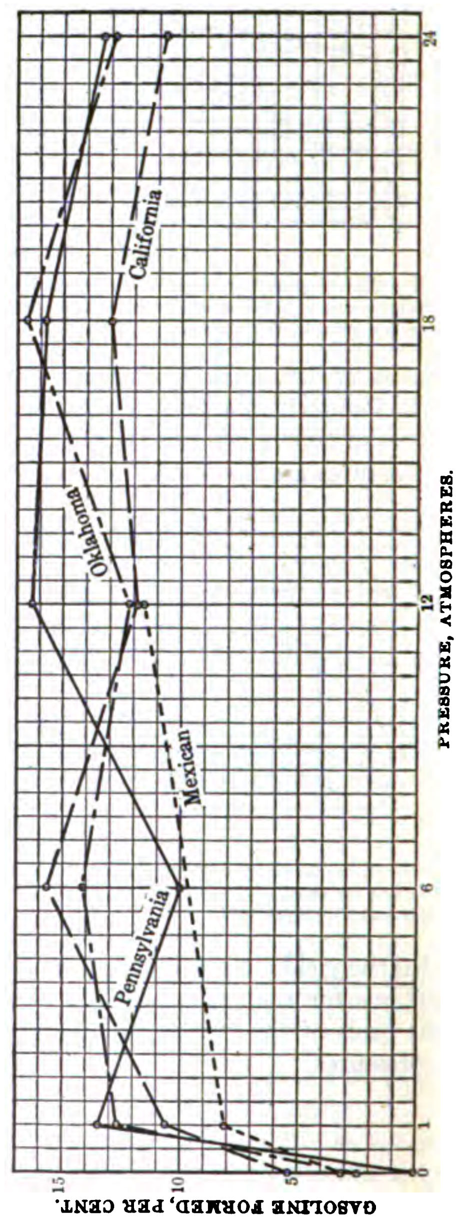

FIGURE 28.—Curves showing percentages of gasoline (hydrocarbons distilling up to 150° C.) formed from cracking four oils under varying pressures at 600° C. Results for higher pressures low, owing to oil consumed in building up pressure.

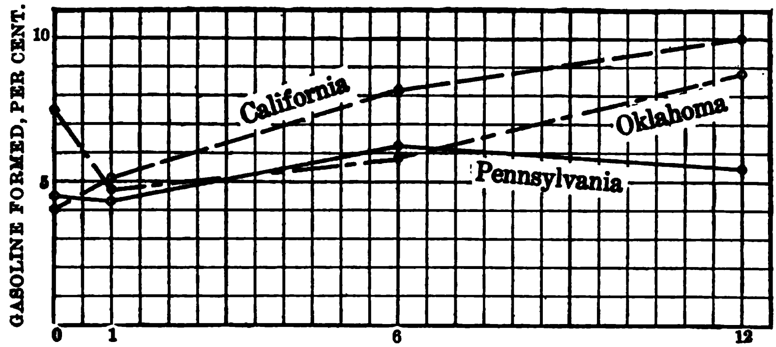

FIGURE 29.—Curves showing percentages of gasoline (hydrocarbons distilling up to 150° C.) formed from cracking three oils under varying pressures at 750° C. Results for higher pressures low, owing to oil consumed in building up pressures.

with sulphuric acid. The effect of pressure on the relative proportions of unsaturated products formed has already received comment. Table 21 showing the data mentioned follows.

TABLE 21.—*Differences in specific gravity of cuts up to 150° C. from oils cracked at like pressures, and temperatures of 600° and 500° C. (Showing differences between gasoline and aromatic hydrocarbons.)*

Pressure.	Difference in specific gravity in oil from—			
	Pennsylvania.	Oklahoma.	California.	Mexico.
Atmosphere.				
(a)	0.009	....	....	....
1	.032	0.046	0.043	0.046
6	.042	.081	.051	....
12	.070	.061	.056	.032
18	.086	....	....	....
24	.088	.111	.053	....

[a] Vacuum.

PROPERTIES OF CRACKED GASOLINE.

As regards the general properties of hydrocarbons, the properties of a gasoline may be largely predicted if its method of production is known. The presence of olefins does not introduce much change in physical properties, but olefin formation, which is bound to occur to some degree when cracked gasoline is produced, is invariably accompanied by the formation of aromatic hydrocarbons, and these increase the specific gravity.

Table 79 (p. 212) shows the relative boiling points and specific gravities of these various hydrocarbons. The data mentioned indicate that one should not expect the same gravity in a cracked gasoline as prevails in a straight distillate gasoline. As previously mentioned, the introduction of cracked gasolines and especially blended gasolines, has caused the use of specific-gravity values as means of identification to be of little value. To learn the character of a gasoline, one must determine other important properties such as distillation range, behavior toward chemical reagents, and explosive energy.

Many persons believe that a cracked gasoline can be made to produce more power than a "straight" distillate gasoline. On the basis of heating value it is at least as valuable. The situation as regards the identification of gasoline is almost directly analogous to that in the illuminating-gas market where the old standard of candlepower largely prevails instead of the more significant basis of heat units.

Because of the increasing demand for gasoline, the user will soon have to take cracked gasoline regardless of his prejudices. As quantities of it are being put on the American market to-day scientific information concerning its constituents and the proper methods of using it seems desirable. Work toward this end is now in progress in the petroleum laboratory of the Bureau of Mines. It is fully expected that satisfactory utilization of cracked gasoline in internal-combustion engines will be obtained with proper admixture of air. In Europe to-day, where the pleasure car is second in consideration to the commercial motor-driven vehicle, a great variety of mixtures are in use, including alcohol, benzene from coal tar, and blended oils—in fact, a variety of materials that were previously considered unfitted for such purposes.

The authors believe that too much stress has been placed on the specific gravity of a gasoline as an index of suitability for a motor fuel, and that as regards efficiency, it is much more important to buy on the basis of the percentage boiling above and below a given temperature. On this basis a cracked gasoline is as good as a gasoline obtained through any other process known to the industry.

One characteristic of a cracked gasoline is slower combustion in an engine. The explosive energy seems to be distributed evenly throughout the entire stroke of the piston, whereas the "straight" distillate tends to develop power much more rapidly. The difference in effect of the two gasolines is particularly noticeable when the automobile containing the engine is being driven up hill.

CONCLUSIONS AS TO GASOLINE FORMATION.

It appears, therefore, that all the products of petroleum from the lightest distillates to the heaviest residuum, including heavy crude oils of an asphaltic nature, such as crude Mexican and California oils, can be successfully cracked and large yields of gasoline hydrocarbons obtained.

It should be noted that the production of gasoline hydrocarbons from a given quantity of original oil is not limited to the first run, but that the residuum above the gasoline fraction can be rerun through the furnace and a like proportion of cracked gasoline obtained. The process may be repeated until the oil has been completely converted. In this manner 50 to 60 per cent of the original oil can be converted into gasoline, as has been repeatedly demonstrated with the small laboratory apparatus used by the authors.

The favorable conditions for gasoline formation seem to be moderate temperatures and high pressures. In commercial work, a temperature ranging between 500° and 550° C. and a pressure of 12 atmospheres and upward will be found suitable, as high a pressure as can be maintained within the limits of safety being desirable, because of the improved quality of the gasoline.

FORMATION OF AROMATIC HYDROCARBONS.

The experiments herein described have served to determine the most favorable conditions for transforming petroleum into aromatic hydrocarbons. It has long been known, as noted in the earlier part of this report, that aromatic hydrocarbons can be produced from certain types of petroleum, but it has never been established, prior to the experiments mentioned, that aromatic hydrocarbons can be produced in considerable quantities from any type of oil, whether the oil has a paraffin, an asphaltic, or a mixed base, or whether it contains appreciable or slight quantities of naphthenes or nitrogen, or any other constituent.

The authors' experiments demonstrated that the type of oil selected for treatment is of relatively little importance as compared with the control conditions and this has been verified in operations on a large scale.

The distinguishing characteristic of aromatic hydrocarbons, as compared with paraffin or olefin hydrocarbons of equivalent boiling points, is their greater density, or higher specific gravity. For example, benzene, which boils at 80.4° C., has a specific gravity of 0.883; and toluene, with a boiling point of 110.3° C., has a specific gravity of 0.872, whereas heptane and octane boil at 98° and 125.5° C. and have specific gravities of 0.708 and 0.712.

DETERMINATION OF AROMATIC HYDROCARBONS IN CRACKED PETROLEUM.

PHYSICAL PROPERTIES CONSIDERED.

In determining the quantitative yields of the aromatic hydrocarbons in the recovered oils the determination of each of the following physical properties was suggested as of possible utility: Solubility, melting point, boiling point, specific gravity, refractive index, surface tension, viscosity, and "molecular weights" (effect on boiling point or freezing point when in solution). If the constituents of a mixture differ as to the first three of these properties, an actual separation may be possible. The other properties can at most serve only as a means of differentiating the constituents.

Surface tension is readily influenced by the presence of small quantities of impurities and, in the absence of definite knowledge concerning its nature, is of little service. "Molecular weights"[a] have been shown to be unreliable even when determined for mixtures that are chemically more homogeneous than those obtained in the present work.

The refractive index is related closely to specific gravity, and on account of that relation did not demand special consideration. Viscosity is not an additive property. In actual fact, information was needed regarding only the first four of the properties listed.

A consideration of actual conditions revealed the following state of affairs: The cracked petroleum oil is a complex mixture containing a series of aromatic hydrocarbons, of which the most important are benzene, toluene, and xylene. "Xylene" represents a mixture of the three isomers, the properties of which are closely related. Higher aromatic hydrocarbons are likewise present, and work is at present in progress for the purpose of studying methods for separating and determining them. In the mixture are also considerable quantities of nonaromatic hydrocarbons of various classes and of almost all boiling points. Thus, it appeared improbable that the desired separation and estimation might be accomplished by making use of any one property.

Separation through differences in boiling point needs little discussion. That benzene, toluene, and xylene can be separated by distillation is indicated by the differences in their boiling points, which are as follows: Benzene, 80.4° C.;[b] toluene, 110.3° C.;[c] xylene, 138° to 142° C.[d]

However, although the three aromatic hydrocarbons may be separated from each other by rectification they can not be isolated from the aliphatic hydrocarbons that are present in the cracked oil and have boiling points approximately the same as those of the benzene, toluene, and xylene. In the determination of the aromatic compounds the wide differences between the specific gravities of the aromatic hydrocarbons and those of the other constituents of approximately like volatility were of great aid. These differences are shown in Table 22.

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[a] Rittman, W. F., and Egloff, Gustav, Relations among the physical constants of petroleum distillates: Jour. Ind. and Eng. Chem., vol. 7, July, 1915, p. 582.

[b] Knops, Carl, Über die Molekularrefraktion der Isomerieen Fumar-Maleinsäure, Mesacon-Citracon-Itaconsäure und des Theophens und ihre Beziehung zur chemischen Constitution diesen Substanzen: Liebig's Annalen, Bd. 248, 1888, p. 175.

[c] Landolt, H., and Jahn, H., Über die Molekularrefraktion einiger einfacher organischer Verbindungen für Strahlen von unendlich grosser Wellenlänge: Ztschr. phys. Chem., Bd. 10, 1892, p. 303.

[d] Barbier, P., and Roux, L., Recherches sur la dispersion dans les composés aromatiques: Bull. Soc. chim. Paris, ser. 3, t. 3, 1890, pp. 255–261.

TABLE 22.—*Differences in specific gravity of paraffin and aromatic hydrocarbons having approximately the same boiling points.*

PARAFFIN HYDROCARBONS.

Hydrocarbon.	Boiling point.	Specific gravity.[a]
	° *C.*	
Dimethyl pentane	86	0.711 at 0° C.
Diisobutyl	108.5	.713 at 0° C.
Nonane	[b] 131.5	.732 at 12.7° C.
AROMATIC HYDROCARBONS.		
Benzene	80.4	0.876 at 20° C.
Toluene	110.3	.871 at 14.7° C.
Xylene (ortho)	142	.885 at 14.1° C.
Xylene (meta)	139	.869 at 15.7° C.
Xylene (para)	138	.866 at 14.7° C.

[a] Determined at temperature shown. [b] Determined at pressure of 751 mm.

The specific gravity of any compound may be easily and accurately determined, and its determination was suggested as a satisfactory method of differentiating between compounds that are alike in the property of volatility.

The use of dimethylsulphate as a solvent has been investigated by Valenta,[a] Perkin and Harrison,[b] Chapin,[c] Reeve and Lewis,[d] Sommer,[e] Church and Weiss,[f] and by Rittman, Twomey, and Egloff.[g] The only logical conclusion to be had from a review of these researches is that this method is not to be recommended.

Owing to the relatively high freezing point of benzene, various investigators have recommended that the benzene content of a given oil be quantitatively estimated by a freezing method.

Some experiments, therefore, were conducted with known mixtures of benzene and unrefined gasoline with a specific gravity of 0.728.

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[a] Valenta, E., Über die Verwendung von Dimethylsulfat zum Nachweis und zur Bestimmung von Teerölen in gemischen mit Harzölen und Mineralölen und dessen Verhalten gegen fette Öle, Terpentinöl, und Pinolin: Chem. Ztg., Jahrg. 30, Mar. 28, 1906, pp. 266-267.

[b] Perkin, F. W., and Harrison, T. W., The action of dimethylsulphate on oils of the aromatic and aliphatic series: The Analyst, vol. 33, January, 1908, pp. 2-11.

[c] Chapin, R. M., Dimethylsulphate test for creosote oils and creosote dips: Circular 167, Bureau of Animal Industry, 1911, pp. 1-7.

[d] Reeve, C. S., and Lewis, R. H., Application of dimethylsulphate test for determining small amounts of petroleum or asphalt products in tars: Jour. Ind. and Eng. Chem., vol. 5, April, 1913, pp. 293-295.

[e] Sommer, Albert, Methods of asphalt examination: Jour. Ind. and Eng. Chem., vol. 2, May, 1910, pp 186.

[f] Church, S. R., and Weiss, J. M., Paraffin bodies in coal-tar creosote and their bearing on specifications: Jour. Ind. and Eng. Chem., vol. 6, 1914, p. 296.

[g] Rittman, W. F., Twomey, T. J., and Egloff, Gustav, Estimation of aromatic hydrocarbons in cracked petroleum: Met. and Chem. Eng., vol. 13, October, 1915, pp. 682-686.

The mixtures were treated at three temperatures. Melting ice gave a temperature of 0° C. A mixture of ice and salt gave a temperature of −20° C. A temperature of −40° C. was obtained by mixing carbon dioxide with an excess of acetone in a Dewar flask.

At 0° C. the only mixture to solidify within a reasonable time of treatment was the 90 per cent mixture, which gave a recovery of 20 per cent. In each experiment the mixture was kept at the required temperature for 15 to 20 minutes with constant stirring. Furthermore, inoculation of the solution yielded no different results. At −20° C. results were obtained as shown in Table 23. At −40° C. results were obtained as shown in Table 24.

These results indicate that benzene crystals tend to include so much mother liquor that the freezing method for determining benzene is unreliable. It appears also that in the presence of an excess of nonaromatic constituents the benzene crystals may be either prevented from separating out or may be dissolved as soon as formed. In any event the freezing method seems to have little to recommend it. The tables (23 and 24) follow.

TABLE 23.—*Percentages of solids formed when benzene-gasoline mixtures were cooled to a temperature of −20° C.*

Test No.	Percentage of benzene in mixture.	Percentage, by volume, solidifying.
1........	90	100
2........	80	100
3........	70	100
4........	60	40
5........	50	25
6........	40	0
7........	30	0
8........	20	0
9........	10	0

TABLE 24.—*Percentages of solids formed when benzene-gasoline mixtures were cooled to a temperature of −40° C.*

Test No.	Percentage of benzene in mixture.	Percentage, by volume, solidifying.
1........	90	100
2........	80	100
3........	70	100
4........	60	100
5........	50	100
6........	40	95
7........	30	40
8........	20	10
9........	10	0

Previous considerations indicated that the most promising physical property on which to base an analytical method was specific gravity. By making use of the differences in gravities of aromatic and nonaromatic hydrocarbons boiling at the same temperatures it was possible to estimate with some accuracy the content of aromatic hydrocarbons. This method required a knowledge of the average specific gravity of nonaromatic constituents, and values were assumed that had been indicated as the result of much experience. In order to check these the aromatic content of various distillates was determined by the standard chemical method of nitration.[a] A close agreement was noted among results obtained by the two methods—the tedious one of nitration and the simple one of specific gravity. The results of the determinations appear in Table 25 and in figure 30

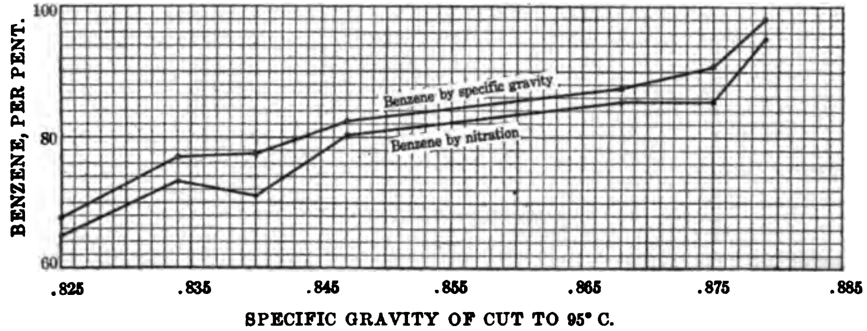

FIGURE 30.—Curves showing agreement of estimated percentages of benzene in cracked-oil distillates by the nitration and by the specific-gravity methods.

TABLE 25.—*Results of estimates of percentage of benzene present in various mixtures by the methods of specific gravity and nitration.*

Specific gravity of sample.	Percentage of benzene present.		Difference between values in columns 2 and 3.
	Estimated on basis of specific gravity.	Estimated on basis of nitration figures.	
1	2	3	4
0.824	65.0	63.6	1.4
.828	68.1	66.2	1.9
.840	75.0	72.0	3.0
.848	80.0	77.2	2.8
.867	91.9	88.0	3.9
.872	95.0	92.3	2.7
.878	98.8	96.0	2.8

In order to verify the results obtained in the course of this investigation four samples were sent to laboratories that specialize in the examination of hydrocarbon mixtures. These laboratories worked independently on samples of a cracked oil and on a distillation cut up to 200° C. obtained from the latter, the distillate representing more than 42 per cent of the cracked oil. The results of the analyses are shown in Table 26.

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[a] Lunge, Georg, Coal tar and ammonia, 4th ed., vol. 2, 1909, p. 765.

TABLE 26.—*Results of analyses of cracked petroleum for benzene and toluene from tests performed independently in various laboratories.*

Analysis No.	Cracked oil.		Distillation cut.	
	Benzene.	Toluene.	Benzene.	Toluene.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
1	10.50	11.50	29.00	26.25
2	11.68	11.33	23.20	26.20
3	....	....	39.91	23.55
4	9.60	6.60	30.20	24.80
5	13.00	10.70	26.20	23.80
Average	11.19	10.03	29.70	24.92

COMMENTS ON ANALYSES.

Analysis 1.—The method employed was distillation, followed by deolefinization and redistillation with subsequent determination of aromatics by the sulphonation method. The results show one slight discrepancy in that they do not indicate the same ratio of benzene and toluene in the cracked oil and in the derived distillation cut.

Analysis 2.—The oils were distilled and redistilled, efficient fractionating columns being used, and subsequent determinations were made by both sulphonation and nitration methods. The latter were given the preference. Results were submitted with the statement that they were probably 1 to 2 per cent too high. Here again there is a discrepancy between the results for cracked oil and those for the distillation cut, and it is in the opposite direction to that noted in analysis 1. The results of both analyses 1 and 2 were probably as reliable as could be obtained by laboratories working in ignorance of the exact conditions under which the cracked oil had been produced and distilled.

Analysis 3.—The analysis was made with a single separation by distillation followed by duplicate determinations by the methods of sulphonation and freezing. The figure for toluene is near to that obtained by other laboratories. That for benzene is high, probably because reliance was placed on the results of the method of freezing.

Analysis 4.—The results were submitted without statement as to the method employed. The results are in general low, indicating a reliance on purely chemical methods.

Analysis 5.—The analysis was made by the method of distillation and specific gravity indicated above. The results are similar to those of analyses 1 and 2, but have the advantage of agreeing as to the ratio of benzene and toluene in the cracked oil and in the distillation cut.

CONCLUSIONS.

The following conclusions are based on the results obtained in this series of experiments which were performed with the degree of care and accuracy generally feasible in commercial laboratories:

Solubility methods for the separation and determination of aromatic compounds in cracked petroleum do not seem to be satisfactory.

Results approximating those of the best chemical methods may be obtained by the method of separating aromatic hydrocarbons with three successive distillations and determining by specific gravity relations their percentage in distillation cuts.

The method of procedure that has seemed most satisfactory on the basis of present experience is as follows:

Seven hundred grams of cracked oil is distilled from a 1-liter distilling flask through a fractionating column whose efficiency is at least as great as that of a 5-inch Hempel column of aluminum beads. Two successive redistillations are conducted, likewise with efficient fractionating columns. For these a 200-gram part of the cut at 175° C. is taken. For the first redistillation, fractions are separated at temperatures of 105°, 130°, and 160° C. For the second the cuts are made at 95°, 120°, and 150° C.

Specific gravities are then determined by whatever method seems convenient. Measurements should be accurate to about one part in a thousand. The percentage of aromatic hydrocarbons is then calculated from these values on the basis of the following gravity relations in the various cuts:

Temperature of cut.	Specific gravity of aromatic hydrocarbon.	Specific gravity of nonaromatic hydrocarbon.
95° C.	0.880	0.720
120° C.	.871	.730
150° C.	.869	.760

The one assumption on which this method is based is the gravity value assigned to the nonaromatic constituents of each distillation cut, but this value need not be known with great accuracy. With present knowledge of the character of the cracking process, it is desirable occasionally to use chemical methods of determination for verifying results obtained on the basis of these specific gravities.

PRESENT APPLICATION OF SPECIFIC GRAVITY METHOD OF DETERMINING AROMATIC HYDROCARBONS.

For the products obtained in the laboratory runs with the small experimental furnace, the yields of recovered oil were too small to permit elaborate fractionation. Consequently, a simpler method was adopted, as follows:

The distillation cuts of the cracked or recovered oils up to 150° C. contain a mixture of the three aromatic hydrocarbons, benzene, toluene, and xylene, whose specific gravities are approximately equal. An average value of 0.875 represents them with sufficient accuracy. For the specific gravities of nonaromatic constituents found in the 150° C. distillation cut, use was made of figures obtained under identical conditions in another series of experiments for the determination of physical constants of crude petroleum and products from various fields. Following are the average specific gravities of 150° C. distillation cuts from the original oils designated:

Pennsylvania oil	0.721
Oklahoma oil	.734
California oil	.742
Mexican oil	.759

These figures, in connection with the specific gravity value for the mixed aromatic hydrocarbons, were used in calculating the percentage formation of low-boiling aromatic hydrocarbons. For example, a California oil (Kern River crude) that was cracked at a temperature of 600° C. and at a pressure of 12 atmospheres gave a percentage recovery of 52.5 per cent. On distillation, 22.5 per cent boiled below 150° C., the specific gravity of the cut being 0.842. The last figure represented a mixture containing 75 per cent of the aromatic hydrocarbons. The total low-boiling aromatic hydrocarbons formed, on the basis of the original oil, were therefore 75 per cent times 22.5 per cent times 52.5 per cent, or 8.3 per cent.

LOW-BOILING AROMATIC HYDROCARBONS FORMED IN EXPERIMENTS.

The values obtained by means of the calculations appear in Table 27 following. Here again an excellent agreement among the determinations for the various original oils is to be noted, demonstrating that the conditions of treatment are of more importance than basic physical and chemical differences.

TABLE 27.—*Results of cracking oils under varying conditions of temperature and pressure, showing percentages of low-boiling aromatic hydrocarbons (combined benzene, toluene, and xylene) formed, calculated on basis of original oil.*[a]

Furnace conditions.		Percentage of low-boiling aromatics formed from original oil from—				
Temperature.	Pressure.	Pennsylvania.	Oklahoma.	California.	Mexico.	Average.
°C.	*Atmospheres.*					
850	1	2.4	....	....	....	2.4
850	(b)	....	4.1	4.4	....	4.3
800	(b)	....	4.6	7.1	....	5.9
750	12	5.6	7.7	8.0	....	7.1
750	6	6.8	5.9	7.9	....	6.9
750	1	5.1	4.7	5.0	....	4.9
750	(b)	4.1	4.2	2.2	2.9	3.4
650	(b)	....	1.4	....	....	1.4
600	24	12.4	11.2	9.7	....	11.1
600	18	13.4	14.0	12.3	....	13.1
600	12	13.4	6.9	8.8	4.1	8.3
600	6	7.1	9.8	12.2	....	9.7
600	1	7.3	5.8	5.1	4.3	5.6
600	(b)	1.0	1.1	3.0	0.4	1.4
550	24	....	4.7	7.7	....	6.2
550	18	....	7.3	....	....	7.3
500	24	7.0	2.1	4.2	....	4.4
500	18	5.6	....	....	....	5.6
500	12	5.6	1.8	3.7	0.9	3.0
500	6	2.6	....	5.4	....	4.0
500	1	1.8	0.6	1.2	1.1	1.2
500	(b)	0.7	....	....	....	0.7
450	18	....	3.3	....	....	3.3
450	12	2.9	1.3	1.1	....	1.8
400	10	....	....	....	0.2	0.2

[a] Results for higher pressures are low, owing to oil being consumed in building up pressure.
[b] Vacuum.

In considering the yields, the reader should remember that, as in gasoline formation, the necessity of building up high pressures, and of superheating a part of the oil in order to obtain this result in minimum time, is responsible for the low yields reported.

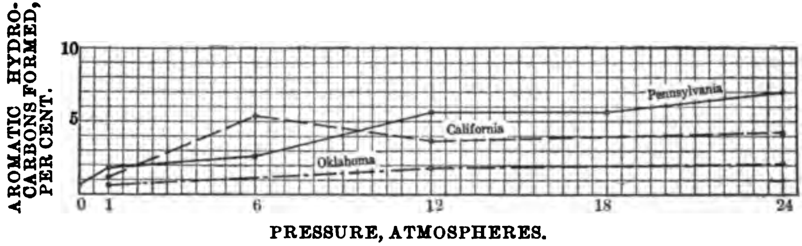


FIGURE 31.—Curves showing percentages of combined aromatic hydrocarbons (benzene, toluene, and xylene) formed from cracking three oils under varying pressures at 500° C. Results for higher pressures low owing to oil consumed in building up pressures.

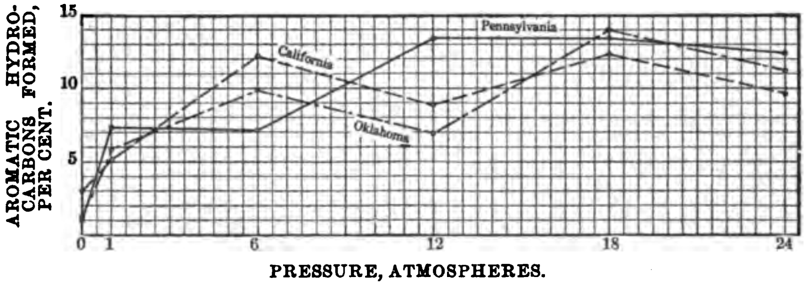


FIGURE 32.—Curves showing percentages of combined aromatic hydrocarbons (benzene, toluene, and xylene) formed from cracking three oils under varying pressures at 600° C. Results for higher pressures low owing to oil consumed in building up pressures.

The higher temperatures are evidently more favorable for the formation of aromatic hydrocarbons than the lower temperatures, and recoveries on the basis of the original oils seem to be greatest at temperatures of 600° to 700° C., as is clearly indicated by the values

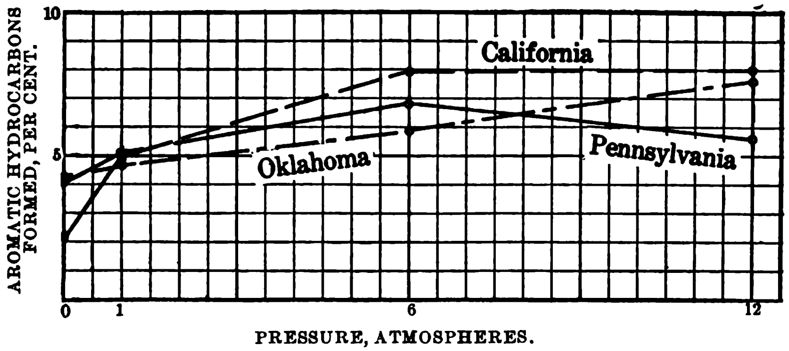


FIGURE 33.—Curves showing percentages of combined aromatic hydrocarbons (benzene, toluene, and xylene) formed from cracking three oils under varying pressures at 750° C. Results for higher pressures low owing to oil consumed in building up pressures.

plotted in figures 31, 32, and 33, which show the percentage yields of the low-boiling aromatics at constant temperatures of 500°, 600°, and 750° C.

Figure 32, which shows the most satisfactory results for the three temperatures plotted, illustrates the close agreement when three oils of widely different characteristics were treated under favorable conditions. At a pressure of 18 atmospheres (270 pounds per square inch) there was an approximate difference of only 1 per cent among the percentages of aromatic hydrocarbon recovered from the oils.

The use of pressure favors the formation of aromatic hydrocarbons, and the general results seem to indicate that a pressure of 10 to 15 atmospheres gives the best results. The relative influences of temperature and pressure are indicated in figure 34. Here the average yields of the three oils at the three temperatures of 500°, 600°, and 750° C. and at pressures of 1 to 24 atmospheres are plotted. The progressive increase in yields with pressure is clearly evidenced.

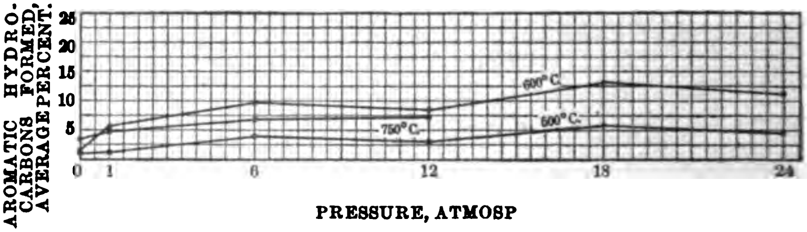


FIGURE 34.—Curves representing average results of cracking oils, showing influence of varying temperatures and pressures on formation of combined aromatic hydrocarbons (benzene, toluene, and xylene). Results for higher pressures low, owing to oil consumed in building up pressure.

Table 28, showing a summary of the results of cracking four different oils under varying conditions of temperature and pressure, is presented below:

TABLE 28.—*Summary of results of cracking oils under varying conditions of temperature and pressure, showing average percentages of aromatic hydrocarbons formed.*[a]

Pressure.	Average percentage of aromatic hydrocarbons formed at temperatures of—							
	450° C.	500° C.	550° C.	600° C.	650° C.	750° C.	800° C.	850° C.
Atmospheres.								
([b])		0.7		1.4	1.4	3.4	5.9	4.3
1		1.2		5.6		4.9		2.4
6		4.0		9.7		6.9		
12	1.8	3.0		8.3		7.1		
18	3.3	5.6	7.3	13.1				
24		4.4	6.2	11.1				

[a] Results for higher pressures low, owing to oil consumed in building up pressure.
[b] Vacuum.

CONCLUSIONS AS TO FORMATION OF AROMATIC HYDROCARBONS.

The data given establish that, as in gasoline formation, all types of petroleum oils, from light distillates to the heaviest residuum, including heavy asphaltic-base oils, can be made to yield aromatic hydrocarbons in commercial quantities. The residuum above the lower-boiling aromatic fraction can be rerun, and the aggregate yields from a given quantity of original oil can be proportionately increased.

The favorable conditions for aromatic formation seem to be high temperatures and moderate pressures. In commercial work a temperature ranging between 625° and 700° C. and a pressure of upward of 8 atmospheres will generally be most suitable.

GENERAL SUMMARY OF LABORATORY EXPERIMENTS.

The results of the experiments described led to the following conclusions:

In procuring the desired products of the cracking reaction, the physical and chemical properties of an original oil are of secondary importance compared with the influence of temperature, pressure, time, and concentration.

Under like conditions, practically identical results have been obtained from five different oils, and the differences reported may be attributed, in part, as much to variation in rate of reaction as to the actual production of dissimilar equilibrium products. One exception noted relates to the production of carbon, a residual product, the formation of which seems to be proportional to the quantity contained in the original oil. Viscosities and specific gravities of the oils obtained show in some measure the influence of the properties of the original oils, but the differences are only slight and can perhaps be explained by the system not having been allowed to reach complete equilibrium.

The experiments have indicated the commercial possibilities and advantages of hydrocarbon reactions carried out in a cracking chamber composed of a vertically arranged tube, when the products to be treated are in a gaseous state and are cracked in an atmosphere of decomposition products. The results obtained with large-scale equipment, subsequently described, have demonstrated the correctness of these conclusions.

AROMATIC HYDROCARBONS USED IN MAKING EXPLOSIVES.

The war in Europe has created an enormous demand for benzene and toluene as raw materials for the manufacture of explosives. Toluene, which normally sells for less than 25 cents a gallon, can not be bought in quantity at present (November, 1915) at $5 a gallon.

Trinitrotoluene has taken first rank among the explosives used for bursting charges because of its low melting point, its safety in handling, its nonacidity, which eliminates corrosion of metallic surfaces in contact with it, and the ease with which the time of its explosion may be controlled.

METHODS OF MANUFACTURING TRINITROTOLUENE.

There are two popular methods of making trinitrotoluene from toluene. The first involves a three-stage operation, the toluene being first converted to the mono derivative, which is treated with a stronger acid to make the dinitrotoluene, and this in turn being subjected to a still stronger acid to make symmetrical trinitrotoluene, known in the trade as TNT. It is claimed that this three-stage procedure increases the yield, also, that the method is the most economical, in that the spent acid from the third nitration can be used for the second-stage nitration and after having been so used is available for making mononitrotoluene from toluene.

The other method, largely used and recommended by English and French explosives manufacturers, involves a one-stage operation in which the toluene is treated with an "oleum," that is, a nitric-sulphuric acid mixture concentrated enough to bring about complete nitration. The disadvantage of this procedure is that the strong acid remaining at the end of the operation is discarded because after one use it is not strong enough to introduce the third nitro group. The advantage claimed is the relatively smaller equipment required for a given output of product. However, the single-stage operation has the further disadvantage, that the starting material must be pure toluene. In view of the fact that much of the American toluene comes from water-gas tar, all of which contains an appreciable proportion of paraffins, this limitation has been considered a serious objection to the general adoption of single-stage nitration in America.

NITRATION OF TOLUENE FROM PETROLEUM.

Early in the development of the processes outlined in this report, the question of the nitration of the resultant benzene and toluene was recognized as demanding solution. The difficulties involved were of the same sort as those incident to the utilization of aromatic hydrocarbons obtained from water-gas tar, which also is derived from petroleum. Popular opinion was to the effect that toluene derived from petroleum could not be nitrated to form a trinitrotoluene as good as that derived from coal tar. It has been proven, however, that it is easily possible to make from toluene derived from petroleum a trinitrotoluene with as high a melting point and as pure as that from toluene derived from any other source.

The problem involved was to make from a toluene derived from cracked petroleum a trinitrotoluene that would meet current specifications. The latter require a melting point higher than 75° C.; the melting of the absolutely pure compound is 80.2° C.

Two general methods of procedure were suggested. The first was to prepare a crude trinitrotoluene and purify it. This, however, can be accomplished only through some process of crystallization, or washing, which requires the use of expensive organic solvents. Such a method is in vogue abroad, but is not considered practicable in this country.

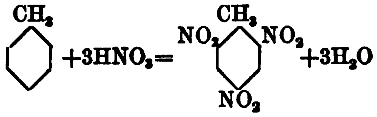
The second method involved the preparation of a trinitrotoluene that should include only such impurities as were soluble in water. To obtain this it was necessary to remove in some stage of the process such impurities as would come through to the end as bodies insoluble in water. The whole problem may be briefly summarized in this simple way: It was necessary to purify one of the following four compounds, toluene, mononitrotoluene, dinitrotoluene, or trinitrotoluene.

Before the most advantageous method of procedure is discussed, it is desirable to consider the properties of the several compounds.

PREPARATION OF TRINITROTOLUENE.

Trinitrotoluene is obtained from toluene by a process of nitration, which may be expressed by the following equation:

$$C_6H_5CH_3+3HNO_3=CH_3C_6H_2(NO_2)_3+3H_2O$$



Water is one of the products of the reaction, and to render it inactive, the nitration is conducted in the presence of sulphuric acid enough to absorb it. The formation of mononitro and dinitro products at intermediate stages is probable, and to fix the nature of the substitution, it is necessary to regulate the conditions of acid concentration under which the reaction occurs. As has been pointed out, the reaction may be brought about in either one, two, or three stages, and with partial or complete purification of the intermediate products.

By starting with chemically pure toluene, it is possible to so conduct the nitration as to obtain directly the trinitro product in such a condition that, by simple washing with water, it may be converted into a product that meets commercial specifications. If, however, one of the paraffin-aromatic mixtures obtained from petroleum is treated in this way the nitroproduct is associated with a brown sludge produced by the action on the paraffins of the strong, hot, acid mixture that is necessarily used. This sludge can not be removed by washing with water, and in fact is separable from trinitrotoluene only with difficulty by any satisfactory commercial method. Strong nitric acid seems to have an effect on paraffins, the nature of which is not exactly known. It is probably a polymerization of some sort, resulting in the formation of resinous bodies.

ISOMERIC FORMS OF NITROTOLUENES.

MONONITROTOLUENE.

Mononitrotoluene ($C_7H_7NO_2$) exists in the three isomeric forms, ortho, meta, and para, of which the physical properties are indicated in the following table:

Physical properties of the three isomeric forms of mononitrotoluene.

Compound.	Melting point.	Boiling point.	Specific gravity.
	°C.	*°C.*	
o-nitrotoluene	−10.5	218	1.168 (15°/15°)
m-nitrotoluene	16.0	231	1.168 (22°/4°)
p-nitrotoluene	54.0	238	1.231 (54°/4°)

Direct nitration of toluene yields a mixture of the three isomers, which, however, contains little of the meta product. This is in accord with the law of substitution in the benzene ring, the methyl group being one that controls the ortho and para positions. It is of importance to note, however, that the relative quantities of ortho and para compounds may be controlled by the conditions under which the nitration is carried out. According to Holleman and Van der Arend,[a] if 1 part of toluene is run into 1.5 parts of nitric acid of a specific gravity of 1.5, at a temperature not exceeding 15° C., the ortho compound is formed chiefly, but if 1 part of toluene is run into 3 of 4 parts of nitric acid of a specific gravity 1.5, and the temperature is maintained at 30° C., the chief product is para-nitrotoluene.

DINITROTOLUENE.

Dinitrotoluene exists in six possible isomeric forms, each of which has been prepared and each of which is solid. These are designated by numbers, the position occupied by the methyl group being 1. The melting point of each is given below:

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[a] Holleman, A. F., and Van der Arend, J. E., Nitration of toluene and para-chlor-toluene: Recueil des Travaux chim. des Pays-Bas, vol. 28, 1909, pp. 408-423.

Melting points of six isomeric forms of dinitrotoluene.

Compound.	Melting point, °C.
2–4	70. 5
3–4	60. 0
2–3	63. 0
2–6	61. 0
3–5	93. 0
2–5	52. 5

The dinitrotoluene produced by the nitration of toluene or of the commercial mixture of ortho and para nitrotoluenes consists chiefly of 2–4 and 2–6 products.

TRINITROTOLUENE.

Trinitrotoluene is theoretically capable of existing in six isomeric forms. Of these the 2–4–6, the 2–3–4, and the 2–4–5 compounds have been prepared. The 2–4–6 or symmetrical, trinitrotoluene is the best known and is by far the chief constituent of the commercial product. The melting points of the three forms mentioned are given below:

Melting points of three isomeric forms of trinitrotoluene.

Compound.	Melting point, °C.
2–4–6	80. 6
2–3–4	112. 0
2–4–5	104. 0

Trinitrotoluene is a pale-yellow solid of absolutely neutral reaction and is unusually stable unless decomposed explosively or burned. It is slightly soluble in water, either hot or cold, but is readily soluble in hot alcohol, though only slightly so in cold.

NITRATION METHOD ADOPTED.

Toluene and mononitrotoluene are liquids having boiling points within limits that permit distillation by ordinary methods. They may be purified by fractional distillation. Dinitrotoluenes and trinitrotoluenes being solids not soluble in water, can be purified only by crystallization from organic solvents. As has been already indicated, the purification of toluene obtained from cracked petroleum seemed to be attended with considerable difficulty. The aromatic compound is invariably associated with both paraffins and olefins of such volatility that distillation does not separate them. Olefins may be removed with sulphuric acid, but the paraffins are not attacked by any reagent that does not affect aromatic hydrocarbons in greater degree.

It therefore seemed desirable to turn to the mononitrotoluene as a medium for purification. Toluene is associated with paraffins and olefins of boiling points in the neighborhood of 110° C. By washing with sulphuric acid, the olefins are extracted. By nitration, toluene may be converted to mononitrotoluene, boiling at 218° to 238° C., whereas paraffins remain unchanged or are in part polymerized to resinous bodies. In the resultant mixture the desired mononitrotoluene is separable from the others by a process of fractional distillation. This separation may be readily accomplished, although distillation in a vacuum or with steam gives the best results. The following scheme of procedure is that which has been found most satisfactory and most economical.

Wash the mixture of toluene, paraffin, olefin, etc., with sulphuric acid of a specific gravity of 1.84 to take out unsaturated hydrocarbons or olefins. A mixture of aromatics and paraffins remains that is saturated with sulphuric acid. The latter does not need removal, as it is one of the reagents involved in the next step. The acid-toluene-paraffin mixture is then treated with a reagent consisting of mixed nitric acid and sulphuric acid strong enough to attack toluene. The concentration of the acid varies with the proportion of paraffins present. The impure mixture obviously requires less acid than would pure toluene. The acid is withdrawn from the nitrated mixture and the latter, after having been washed with water, is distilled in a vacuum or with steam, air distillation being undesirable on account of its producing a certain degree of decomposition.

RESULTS AND BEARING OF NITRATION EXPERIMENTS.

Actual experiments along the lines just indicated have been conducted in the Bureau of Mines laboratory. By the simple process of deolefinization with sulphuric acid, nitration of the unwashed product, and a single vacuum distillation samples of mononitrotoluene have been prepared that on analysis showed nitrogen contents within 0.5 per cent of the theoretical. Yields in laboratory experiments are necessarily lower than can be obtained under commercial conditions, but even here results have been obtained that show a recovery of pure product of 75 to 82 per cent of the total theoretically possible, comparing favorably with the recovery of toluene from coal tar.

The simplicity of this procedure indicates an advantage of petroleum over coal tar as a source of toluene. The product from coal tar may, it is true, be obtained in a pure state, but the processes are complicated and expensive owing to the necessity of removing oxygen and sulphur bodies derived from coal. The toluene derived from tar must be given extensive treatment both by distillation and by washing with chemicals before it can be utilized in nitration processes.

Petroleum-derived toluene is not so easily obtainable in a pure state, but its impurities are not of such nature as to cause trouble when the process of nitration is carried out. In short, there are two simple processes for deriving toluene from petroleum and one difficult process for deriving it from coal tar.

If the operation of nitration is conducted on a large scale, the purity of the mononitrotoluene obtained is highly satisfactory, and makes what has been considered a serious objection a real advantage, in that it is easy to use any petroleum-derived toluene without regard to its state of purity. Instead of attempting to obtain pure toluene at the outset the effort to reach a state of chemical purity is made at the mononitrotoluene stage.

Petroleum-derived toluene is partly purified by distillation, and then mononitrotoluene is prepared, which is completely purified by a distillation with steam or in a vacuum.

The actual preparation of trinitrotoluene from petroleum-derived mononitrotoluene has been accomplished by a modification of the two-stage process in use by one of the large explosive companies of this country. The products obtained, after having been washed with water, satisfied the present customary specification of a melting point above 75° C. The yields have approximated 75 per cent of the yields theoretically possible. Here, again, the factor of yield has not been studied with the care bestowed on quality, as laboratory figures can not at best compare with those obtained in commercial operations.

NITRATION OF BENZENE BY METHOD APPLIED TO TOLUENE.

The utilization of benzene obtained from cracked petroleum has not yet been studied with the care bestowed on the utilization of toluene. Experiments for the preparation of picric acid are now in progress. Nitrobenzene has been obtained by a method identical with that involved in the preparation of nitrotoluene. From such nitrobenzene an 85 per cent yield of aniline oil has been obtained, the latter being one of the more important raw materials in the dyestuff industry.

The general conclusion is that the difficulty of utilizing aromatic hydrocarbons derived from petroleum is slight, requiring only necessary deviations from the conventional methods applied to the carefully purified products obtained from coal.

EXPERIMENTS TO DETERMINE COURSE OF REACTION AMONG AROMATIC HYDROCARBONS.

The series of experiments dealing with general transformations was accompanied by studies that went more into detail regarding the specific products obtained by the cracking reaction. The following considerations have proven of the greatest importance in the present study and accordingly are given separate and detailed discussion.

KINETIC CONSIDERATIONS.

The cracking or decomposition of organic compounds by heat is generally explained on the basis of the occurrence of intermolecular collisions of such violence that the bonds holding together the various atoms or groups are ruptured. This kinetic theory seems to take account of all the phenomena of the reaction. The degree of cracking varies in the same direction as the temperature to which the substance is heated; that is, the higher the temperature the greater is the cracking. This is as would be expected when it is remembered that the velocity of molecular vibration, and consequently the violence of impact, increases with temperature.

Likewise it is known that, other factors being equal, cracking is more easily produced in compounds of large than of small molecular weight. If velocities of vibration are equal, then the large molecule has a larger momentum than the smaller, and at the instant of collision is subjected to a greater strain.

Molecular structure is another factor having an influence that can be largely predicted on the basis of kinetic considerations. Cyclic compounds are more stable than those of the straight-chain type, as would be expected in view of their greater compactness of structure. Likewise if a compound consists of a cyclic nucleus with an attached side chain, or of two cyclic nuclei united through a straight chain, the weak point is undoubtedly the chain. As indicated later, there is a general tendency for decomposition of petroleum up to a certain stage to result in the production of benzene, which is the simplest and most stable of the monocyclic hydrocarbons.

With the preceding considerations in view it is easy to understand the character of the cracking process as indicated by the information already at hand. It may be stated also that the indications obtained from the equilibrium constants, figured according to the Nernst equation (see Table 4), are entirely in accord with those just mentioned.

Some important indications may be obtained regarding relative degrees of formation of various aromatic hydrocarbons without considering the absolute conditions of cracking. Experience has indicated that large paraffin molecules are less stable than small ones, that paraffins are less stable than olefins, and that stability increases with decrease in saturation. It appears, therefore, that benzene formation requires a more strenuous reaction in the cracking process than does toluene formation, as toluene is in part aliphatic, owing to the presence of the methyl group that makes up the side chain. Likewise it appears that xylene, having two methyl groups, should show the same variation in stability as toluene but that it should always occur in about half the quantity of toluene. The probability of its formation is half that of the formation of toluene and the probability of its decomposition twice as great.

The formation of naphthalene and anthracene seems to necessitate the decomposition of benzene nuclei. The formation of these polycyclic hydrocarbons should, therefore, be coincident with a decrease in the number of monocyclic molecules.

EXPERIMENTS WITH INTERAROMATIC REACTIONS.

Both scientifically and commercially the differences among the various aromatic hydrocarbons are of almost, if not quite, as great importance as the differences among classes. Two series of experiments were, therefore, conducted for the purpose of establishing facts concerning the influence of varying conditions of temperature and pressure on interaromatic reactions. In the first series four typical monocyclic hydrocarbons, cymene, xylene, toluene, and benzene, were subjected to cracking, each at three different temperatures, and at each temperature under four pressures. The solid polycyclic hydrocarbons, naphthalene and anthracene, were also cracked, but on account of experimental difficulties the results obtained were only qualitative. In the second series petroleum was employed as the original material. A radically different scheme of procedure was used, but the results agreed entirely with those of the first series.

A study of reaction products has furnished indications that may be generally summarized as follows:

The course of the reaction is in the direction of decrease in size of molecule when the degree of saturation is unchanged.

Reactions may occur in the direction of dehydrogenation either with increase or with decrease in size of molecule.

Practically no reverse reactions occur.

WORK OF OTHER INVESTIGATORS.

More or less work has been done by several previous investigators on the problem of thermal reactions of aromatic compounds, but no one has made a systematic study of such reactions. The information desired at present concerns the influence of conditions of temperature and pressure on quantitative and qualitative relations among end products and this is not adequately supplied by the literature. The following brief abstracts deal with experiments that seem most important in the present connection.

The classic researches of Bertholet[a] have furnished a foundation for the knowledge in this general field. His series of pyrogenic reactions is comprehensive and the results obtained are valuable, though not such as to meet the needs under consideration.

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[a] Bertholet, M., Action de la chaleur sur quelques carburés d'hydrogène, Théorie des corps pyrogénés; Ann. chim. phys., ser. 4, t. 9, 1866, pp. 445–483; Des carburés pyrogénés, Ann. chim. phys., ser. 4, t. 12, 1867, pp. 5–96, t. 16, 1869, pp. 143–187.

Ferko[a] furnished some valuable information, particularly with regard to the direction of reactions. He passed the vapors of several hydrocarbons, either pure or mixed with ethylene gas, through an iron tube heated over a 40-cm. length in a gas furnace. The products were condensed and their composition studied. The more important of the results obtained by Ferko are summarized in Table 29 following.

TABLE 29.—*Summary of results obtained by Ferko in his experiments on the transformation obtained by passing various original hydrocarbons through a tube heated to "red heat."*

Original material.		Reaction products.								
Hydrocarbon.	Quantity.	Benzene.	Toluene.	Styrolene.	Ethyl-benzene.	Di-naphthalene.	Diphenyl.	Naphthalene.	Phenanthrene.	Anthracene.
	Grams.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
Benzene and ethylene	1,320	6.0	0.0	1.3	0.0	0.0	22.7	0.0	0.8	1.1
Toluene	1,300	11.5	13.8	.5	.0	.0	2.1	3.0	1.1	1.0
Toluene and ethylene	1,300	15.4	12.3	.8	.0	.0	.0	.0	.0	1.5
Naphthalene and ethylene	900	.0	.0	.0	.0	13.3	.0	44.5	.1	.0
Ethyl-benzene	500	15.0	1.0	2.0	4.0	.0	.6	2.2	2.6	.4

The table shows that no reaction products were obtained that were not in the order of decrease of saturation or of molecular weight if the saturation remained unchanged. Thus benzene yielded no toluene nor any other compound of equal saturation and of greater molecular weight. Naphthalene yielded neither benzene nor toluene. From ethyl-benzene, however, it was possible to obtain toluene, benzene, and compounds of lower saturation. The work of Ferko seems to have been the most valuable of all hitherto done in this field, but it gives little attention to the influence of temperature and none to that of pressure.

Haber[b] studied the pyrogenic reactions of various substances, among them benzene, at temperatures ranging between 600° and 800° C. and 900° and 1,000° C. One of his observations is contrary to the results of the authors' experiments. He states that although benzene goes readily to diphenyl it does not form naphthalene.

McKee,[c] as a result of some qualitative experiments, has concluded that the order of hydrocarbon decomposition is as follows: Higher paraffins → lower paraffins → olefins → acetylenes → benzene and homologues → diphenyl → naphthalene, etc., → tarry matter → carbon and gas. In McKee's experiments benzene was passed through a hot copper tube and the degree of decomposition determined by the change in the specific gravity of the product. No analysis or separation of the product seems to have been attempted.

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[a] Ferko, Paul, Über einige pyrogéné Reactions: Ber. Deut. chem. Gesell., Jahrg. 20, Bd. 1, 1887, pp. 660–664.

[b] Haber, F., Theorie der pyrogen reactionen aliphatischer Kohlenwasserstoffe: Ber. Deut. chem. Gesell., Jahrg. 29, Bd. 3, 1896, pp. 2691–2700.

[c] McKee, G. W., On the decomposition of benzene at high temperatures: Jour. Soc. Chem. Ind., vol. 23, Apr. 30, 1904, pp. 403–404.

Smith and Lewcock [a] passed benzene through an iron tube, and at 800° C. obtained a 25 per cent yield of diphenyl. They were not, however, able to reverse the reaction, a condition that they attributed to experimental difficulties. They observed that the quantity of diphenyl produced increased with the rate of feed, indicating that diphenyl is not an equilibrium product, but simply an intermediate one.

EXPERIMENTS WITH PURE AROMATIC HYDROCARBONS AS STARTING MATERIAL.

In the experiments described in this report, cracking was produced in a system having a single (gaseous) phase. The characteristics and advantages of this method of procedure have already been discussed in detail.

Equilibrium relations for some of the reactions involved may be found in Table 4, which contains values figured according to the Nernst approximation formula.

The scheme of procedure followed in the experiments under consideration was, with slight exceptions, identical with that described for the other series. One modification in the making of the "run" is worthy of mention. In the earlier series of experiments pressures were built up in the cracking furnace by the decomposition of some of the oil used in the run. This method introduced a source of error, which, in the more recent experiments, it has been possible to eliminate. Before the beginning of each pressure run the system was connected with a compressor and with natural gas, which was pumped in until the gage gave the proper reading. The compressor was then shut off by closing a valve and the run started. Natural gas was used because it is largely methane, which is one of the end products of all cracking reactions.

For all the liquid hydrocarbons the original material was allowed to flow into the furnace at the rate of about 6 c.c. per minute. When the solid hydrocarbons naphthalene and anthracene were used as initial material it was, of course, necessary to keep them in a molten condition during the run by applying heat to the feed cup. Even then considerable experimental difficulty was involved and no attempt was made to obtain results of more than qualitative accuracy.

The supplementary determinations were made with quantities of recovered oil too small to permit separation of pure products. A single analytical distillation was made through an 8-inch Hempel column, and cuts were made at 100°, 125°, 150°, and 200° C. The first cut was taken as representative of benzene, the second of toluene, the third of xylene, and the fourth of cymene. Above 200° C. the distillation was continued with a shorter fractionating column, and cuts were made at intervals of 50° C. The distillates were chilled and examined for the separation of solid naphthalene and anthracene. On account of the smallness of the quantities obtained no attempt was made to obtain quantitative results for the solid products.

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[a] Smith, Clarence, and Lewcock, William, Pyrogenic decomposition—Benzene: Jour. Chem. Soc., vol. 101, pt. 2, 1912, pp. 1453-1459.

MATERIAL USED IN EXPERIMENTS.

The benzene used in the experiments was the purest commercially obtainable. The sample used was particularly good, as 98 per cent distilled between 80° and 81° C. Its specific gravity was 0.879 (15°/15°).[a]

The toluene used was that sold as "chemically pure." Its specific gravity was 0.869 (15°/15°), and a distillation yielded the following figures:

Results of distillation of toluene used in experiments.

Temperature, ° C.	Cut, per cent.
98	First drop.
98 to 100	0.2
100 to 110	92.0
110 to 115	4.0

The xylene used was also the so-called chemically pure commercial product. Its gravity was 0.862 (15°/15°), and its distillation showed the following results:

Results of distillation of xylene used in experiments.

Temperature, ° C.	Cut, per cent.
88	First drop.
88 to 120	0.7
120 to 137[b]	99.3

The cymene used was also a commercial product, having a specific gravity of 0.863 (15°/15°). The results of its distillation were as follows:

Results of distillation of cymene used in experiments.

Temperature, ° C.	Cut, per cent.
165	First drop.
170 to 179	90

GENERAL RESULTS OF EXPERIMENTS.

The data obtained in the experiments under consideration appear in Tables 30 to 35, which show the scope of the work and indicate the nature of the measurements that were made.

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[a] Specific gravity at 15° C. compared with that of water at 15° C.
[b] Flask dry.

Results are recorded in terms of percentage by volume except when either the initial material or the reaction product was a solid. In such instances mass relations are given.

It will be noted that for the four liquid hydrocarbons, cymene, xylene, toluene, and benzene, the results of the experiments are complete. Quantitative results for the distillations are given for cuts up to 200°, and above that temperature the presence or absence of naphthalene and anthracene is indicated by a plus (+) or a minus (−) sign. For the experiments in which naphthalene or anthracene was the initial material no distillates were obtained below 200° C., and all the results, therefore, are qualitative. In Tables 30 to 35, the results for each test are average number of runs at the stated temperature and pressure. The tables follow:

TABLE 30.—*Results of cracking cymene under various conditions of temperature and pressure.*

Temperature, ° C	650	650	650	650	725	725	725	725	800	800	800	800
Pressure, atmospheres	(a)	1	12	18	(a)	1	12	18	(a)	1	12	18
Oil used, c. c	500	600	600	600	500	400	600	600	500	500	600	600
Oil recovered, per cent	83.0	62.0	53.3	40.8	52.0	40.0	27.5	25.0	49.0	17.6	15.8	00.0
Specific gravity of recovered oil	0.895	0.921	0.924	0.916	0.927	0.982	0.945	0.934	0.951	0.995	0.926	....
Carbon produced, per cent	1.8	1.0	10.2	16.3	2.4	3.6	37.4	32.8	1.3	9.0	20.8	00.0

RESULTS OF DISTILLATION.

First drop at (° C.)	97		69		80		70		97		....		75		....		73		....		....		....	
Temperature.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.
° C.																								
0 to 100	0.2	......	9.2	0.877	12.0	0.876	15.1	0.875	0.2	......	3.7	......	10.2	0.881	3.8	0.872	1.5	......	1.6	0.872	3.8	0.865	0.0	......
100 to 125	14.5	0.869	2.5	.874	2.7	.875	1.9	.873	3.9	0.878	7.4	0.876	2.3	.870	5.0	.877	4.7	0.874	1.9	.874	1.3	.868	.0	......
125 to 150	17.8	.872	5.9	.876	4.0	.874	4.9	.867	8.9	.880	5.2	.885	2.1	.870	2.8	......	9.5	.882	1.6	.878	1.6	......	.0	......
150 to 200	34.5	.878	21.6	.899	21.3	.874	10.6	.873	17.0	.888	5.4	.898	3.0	.898	7.5	.870	14.5	.895	1.6	......	5.7	.880	.0	......
Above 200:																								
Naphthalene[b]	−	......	−	......	+	......	+	......	−	......	+	......	+	......	−	......	−	......	−	......	−	......	−	......
Anthracene[b]	−	......	−	......	−	......	+	......	−	......	−	......	−	......	−	......	−	......	−	......	−	......	−	......

[a] Vacuum.

[b] Presence (or absence) shown by plus (or minus) sign.

TABLE 31.—*Results of cracking xylene under various conditions of temperature and pressure.*

Temperature, °C.	650	650	650	650	725	725	725	725	800	800	800	800
Pressure, atmospheres	(a)	1	12	18	(a)	1	12	18	(a)	1	12	18
Oil used, c. c.	500	500	600	600	500	500	600	600	500	590	600	425
Oil recovered, per cent.	95.0	69.0	66.0	64.3	72.0	60.0	54.2	46.6	66.0	27.1	31.7	11.5
Specific gravity of recovered oil	0.879	0.905	0.914	0.899	0.888	0.906	0.911	0.900	0.912	0.923	0.904	0.945
Carbon produced, per cent.	0.9	1.5	....	4.4	6.0	....	13.6	25.8	.5	25.0	39.5	38.4

RESULTS OF DISTILLATION.

First drop at (°C.)	77		80		60		....		68		82		65		98		87		77		68		....	
Temperature.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.
°*C.*																								
0 to 100	0.3	......	4.1	0.868	3.8	0.855	0.6	......	0.0	......	2.0	......	3.2	0.857	0.0	......	0.7	......	8.3	0.878	9.1	0.868	5.4	0.875
100 to 125	2.6	......	15.4	.870	18.7	.867	9.2	0.858	9.2	0.861	34.5	0.862	15.2	.869	12.6	0.872	18.5	0.874	7.2	.874	8.6	.867	.8	.875
125 to 150	80.0	0.868	37.7	.870	31.7	.868	45.0	.856	52.5	.862	40.0	.877	27.0	.862	26.0	.869	35.6	.872	4.5	.874	7.9	.864	1.1	.875
150 to 200	1.9	......	2.2	.911	.7	......	.6	......	2.4	......	6.0	.899	.0	......	1.6	......	2.3	......	.9	......	......	......	1.5	.875
Above 200:																								
Naphthalene[b]	−	......	−	......	−	......	+	......	−	......	+	......	−	......	+	......	−	......	−	......	−	......	−	......
Anthracene[b]	−	......	−	......	−	......	−	......	−	......	+	......	−	......	−	......	+	......	−	......	−	......	−	......

[a] Vacuum.

[b] Presence (or absence) shown by plus (or minus) sign.

TABLE 32.—*Results of cracking toluene under various conditions of temperature and pressure.*

Temperature, °C.	650	650	650	650	725	725	725	725	800	800	800	800
Pressure, atmospheres	(a)	1	12	18	(a)	1	12	18	(a)	1	12	18
Oil used, c. c.	500	600	480	600	500	600	600	475	500	600	600	600
Oil recovered, per cent.	98.0	98.3	94.8	60.8	85.0	64.2	48.3	18.0	66.0	32.5	34.2	11.7
Specific gravity of recovered oil	0.872	0.894	0.933	0.912	....	0.924	0.918	0.929	0.903	0.931	0.925	0.927
Carbon produced, per cent.	0.0	1.0	4.7	5.9	1.4	1.3	20.1	34.0	1.8	40.0	24.3	23.4

RESULTS OF DISTILLATION.

Temperature.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.
First drop at (°C.)	....		66		88		82		80		76		....		73		....		....		....		....	
°*C.*																								
0 to 100	0.0	......	3.9	......	21.8	0.872	18.2	0.877	2.1	......	17.3	0.874	30.7	0.883	10.4	0.879	5.6	......	17.6	0.880	16.4	0.866	3.0	0.874
100 to 125	91.6	0.871	85.0	0.875	55.0	.868	31.0	.874	70.5	0.870	34.0	.873	7.5	.870	2.7	......	49.5	0.872	6.6	.879	7.5	.873	4.0	.874
125 to 150	3.4	......	.2	......	.0	......	.0	......	4.2	......	.0	......	.7	......	.0	......	.0	......	.0	......	1.2	......	1.6	......
150 to 200	......	......	.5	......	.0	......	.0	......	.2	......	2.6	......	1.2	......	5.0	......	.1	......	.0	......	.0	......	.7	......
Above 200:																								
Naphthalene [b]	+	......	+	......	−	......	+	......	+	......	+	......	+	......	−	......	+	......	−	......	+	......	−	......
Anthracene [b]	−	......	−	......	−	......	−	......	−	......	−	......	+	......	−	......	+	......	−	......	+	......	−	......

[a] Vacuum.

[b] Presence(or absence) shown by plus (or minus) sign.

TABLE 33.—*Results of cracking benzene under various conditions of temperature and pressure.*

Temperature, ° C.	650	650	650	650	725	725	725	725	800	800	800	800
Pressure, atmospheres	(a)	1	12	18	(a)	1	12	18	(a)	1	12	18
Oil used, c. c.	500	600	600	335	500	500	600	600	500	500	600	600
Oil recovered, per cent.	90.0	86.6	87.5	73.1	94.0	90.0	58.3	53.3	87.0	62.0	22.5	18.3
Specific gravity of recovered oil	0.896	0.898	0.941	0.921	0.904	0.945	0.946	0.923	0.906	0.959	0.922	0.928
Carbon produced, per cent	0.2	0.9	1.5	11.9	1.0	1.4	15.3	34.0	2.5	1.6	6.3	35.0

RESULTS OF DISTILLATION.

First drop at (°C.)	64		69		69		76		76		65		70		71		77		76		68		64	
Temperature.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.
° C.																								
0 to 100	78.5	0.881	66.5	0.885	59.4	0.886	57.0	0.878	77.5	0.888	61.5	0.815	39.7	0.886	42.3	0.885	63.0	0.883	35.6	0.886	16.1	0.888	12.1	0.884
100 to 125	1.6	.882	.0	......	.1	......	.4	......	1.4	.864	.9	......	.0	......	.0	......	.9	......	.3	......	.0	......	.0	......
125 to 150	.8	......	.0	......	.0	......	.0	......	1.4		.7	......	.0	......	.0	......	1.7	......	.5	......	.0	......	.0	......
150 to 200	.4	......	1.2	......	.0	......	.0	......	2.8	.906	.7	......	.0	......	.0	......	6.9	......	3.0	......	.0	......	.0	......
Above 200:																								
Naphthalene [b]	+	......	+	......	+	......	+	......	+	......	−	......	+	......	+	......	+	......	+	......	+	......	+	......
Anthracene [b]	+	......	−	......	−	......	−	......	−	......	−	......	+	......	+	......	−	......	−	......	−	......	−	......

[a] Vacuum.

[b] Presence (or absence) indicated by plus (or minus) sign.

TABLE 34.—*Results of cracking naphthalene under various conditions of temperature and pressure.*

Temperature, °C	650	650	650	725	725	725	800	800	800
Pressure, atmospheres	(a)	1	12	(a)	1	12	(a)	1	12
Material used, grams	400	400	432	400	414	384	400	500	427
Material recovered, per cent	76.0	65.7	30.0	66.4	65.4	....	85.9	80.8	19.8
Carbon produced, per cent	0.0	3.3	21.0	3.5	7.0	40.0	9.5	13.6	5.0

QUALITATIVE RESULTS OF DISTILLATION.

First drop at (°C.)	205	200	205	200	210	....	....	....	....
Naphthalene [b]	+	+	+	+	+	+	+	+	+
Anthracene [b]	−	−	−	+	+	+	+	+	+

[a] Vacuum.

[b] Presence (or absence) indicated by plus (or minus) sign.

TABLE 35.—*Results of cracking anthracene under various conditions of temperature and pressure.*

Temperature, ° C	650	725
Pressure, atmosphere	1	(a)
Material used, grams	125	125
Material recovered, per cent	63.2	56.0
Carbon produced, per cent	18.0	22.0

QUALITATIVE RESULTS OF DISTILLATION.

First drop at (° C.)	300	....
Naphthalene [b]	−	−
Anthracene [b]	+	+

[a] Vacuum.

[b] Presence (or absence) indicated by plus (or minus) sign.

RECOVERIES OF LIQUID PRODUCTS.

A first point to be considered is the yield of liquid reaction products obtained when the hydrocarbons were cracked under various conditions of temperature and pressure. This percentage represents the total of liquid products and of solid products soluble therein, except carbon and gas. The figures given are representative in a way of the stability of the various compounds, their resistance to conversion into carbon and gas being indicated by the proportion of liquid product obtained.

It appears that as regards the four liquid hydrocarbons the order of decreasing stability is benzene, toluene, xylene, and cymene. This is as would be expected, on the theory that with similar molecular forms the more unwieldy units of matter are the more easily broken down.

The yield decreased with increase in temperature and in general with increase in pressure. Table 36, showing the recoveries of liquid reaction products, follows:

TABLE 36.—*Results of cracking four aromatic hydrocarbons under various conditions of temperature and pressure, showing percentages of liquid reaction products recovered.*

Hydrocarbon cracked.	Temperature.	Percentage of liquid reaction product recovered at pressures of—				Average percentage recovered.
		Vacuum.	1 atmosphere.	12 atmospheres.	18 atmospheres.	
	°*C.*					
Cymene	650	83.0	62.0	53.3	40.8	38.8
Do.	725	52.0	40.0	27.5	25.0	
Do.	800	49.0	17.6	15.8	.0	
Xylene	650	95.0	69.0	66.0	64.3	54.2
Do.	725	72.0	60.0	54.2	46.6	
Do.	800	66.0	27.1	31.7	11.5	
Toluene	650	98.0	98.3	94.8	60.8	59.3
Do.	726	85.0	64.2	48.3	18.0	
Do.	800	66.0	32.5	34.2	11.7	
Benzene	650	90.0	86.6	87.5	73.1	65.5
Do.	725	94.0	90.0	58.3	53.3	
Do.	800	87.0	62.0	22.5	18.3	

ORIGINAL HYDROCARBONS RECOVERED.

The percentage of original product recovered is another index of the stability of the compound cracked. It represents total resistance to transformation, whereas the recovery figures for liquid products represent resistance to conversion into carbon and gas. It appears that the order of stability is the same as that indicated in Table 36, but that there are greater differences. The effects of temperature and pressure are analogous to those indicated in the earlier table. Table 37, showing the percentages of original hydrocarbons recovered, is presented below:

TABLE 37.—*Results of cracking four aromatic hydrocarbons under various conditions of temperature and pressure, showing percentages of original hydrocarbons recovered.*

Hydrocarbon cracked.	Temperature.	Percentage of original hydrocarbon recovered at pressures of—				Average percentage recovered.
		Vacuum.	1 atmosphere.	12 atmospheres.	18 atmospheres.	
	°*C.*					
Cymene	650	34.5	21.6	21.3	10.6	11.9
Do.	725	17.0	5.4	3.0	7.5	
Do.	800	14.5	1.6	5.7	.0	
Xylene	650	80.0	37.7	31.7	45.0	30.6
Do.	725	52.5	40.0	27.0	26.0	
Do.	800	35.6	4.5	7.9	1.1	
Toluene	650	91.6	85.0	55.0	31.0	37.1
Do.	725	70.5	34.0	7.5	2.7	
Do.	800	49.5	6.6	7.5	4.0	
Benzene	650	78.5	66.5	59.4	57.0	50.7
Do.	725	77.5	61.5	39.7	42.3	
Do.	800	63.0	35.6	16.1	12.1	

BENZENE FORMATION.

Table 38 following shows the percentages of benzene formed under varying conditions of temperature and pressure. It appears from the table that benzene is formed in appreciable quantity from each of the three hydrocarbons, cymene, xylene, and toluene. The figures in Tables 30 to 35 indicate that neither benzene nor any of the other three monocyclic compounds is produced by the cracking of naphthalene or anthracene.

Table 38 shows that optimum conditions for benzene formation are represented by the temperatures 650° and 725° C., and that pressure is favorable up to a certain limit which appears to be somewhere between 1 and 12 atmospheres. The runs at 18 atmospheres in general showed decreases in the amount of benzene produced. This seems to indicate that at some pressure between 1 and 12 atmospheres a sufficient concentration was attained to permit the hydrocarbons to reach a state of equilibrium during their passage through the furnace and that the higher pressures were unfavorable for benzene production, as is indicated by the volume changes occurring in the reactions.

TABLE 38.—*Results of cracking four aromatic hydrocarbons under various conditions of temperature and pressure, showing percentages of benzene formed.*

Hydrocarbon cracked.	Temperature.	Percentage of benzene formed at pressure of—				Average percentage recovered.
		Vacuum.	1 atmosphere.	12 atmospheres.	18 atmospheres.	
	° C.					
Cymene	650	0.2	9.2	12.0	15.1	5.1
Do	725	.2	8.7	10.2	3.8	
Do	800	1.5	1.6	3.8	.0	
Xylene	650	.3	4.1	3.8	.6	3.1
Do	725	.0	2.0	3.2	.0	
Do	800	.7	8.3	9.1	5.4	
Toluene	650	.0	8.9	21.8	18.3	12.3
Do	725	2.1	17.3	30.7	10.4	
Do	800	5.6	17.6	16.4	3.0	
Benzene	650	78.5	66.5	59.4	57.0	50.7
Do	725	77.5	61.5	39.7	42.3	
Do	800	63.0	35.6	16.1	12.1	

TOLUENE FORMATION.

The figures on toluene formation are shown in Table 39 following. Toluene may be formed from both xylene and cymene, being more readily formed from the former. The amount that can be produced from benzene is negligible, and none can be produced from naphthalene and anthracene. The effects of temperature and pressure seem rather irregular, possibly on account of experimental error. The general tendency seems to have been for maximum yields to occur with decreasing temperature as pressure increases. Thus, in the xylene figures a maximum appears at 800° C. for vacuum runs, at 725° C. for runs at 1 atmosphere, and 650° C. for runs at 12 atmospheres. Beyond this point the curve seems to swing the other way. A like tendency will be noted among cymene figures if the obviously wrong bracketed value is omitted from consideration.

TABLE 39.—*Results of cracking four aromatic hydrocarbons under various conditions of temperature and pressure, showing percentage of toluene formed.*

Hydrocarbon cracked.	Temperature.	Percentage of toluene formed at pressure of—				Average percentage formed.
		Vacuum.	1 atmosphere.	12 atmospheres.	18 atmospheres.	
	°C.					
Cymene	650	[14.5]	2.5	2.7	1.9	4.0
Do	725	3.9	7.4	2.3	5.0	
Do	800	4.7	1.9	1.3	.0	
Xylene	650	2.6	15.4	18.7	9.2	11.4
Do	725	9.2	34.5	15.2	12.6	
Do	800	18.5	7.2	8.6	.8	
Toluene	650	91.6	85.0	55.0	31.0	37.1
Do	725	70.5	34.0	7.5	2.7	
Do	800	49.5	6.6	7.5	4.0	
Benzene	650	1.6	.0	.1	.5	.5
Do	725	1.4	.9	.0	.0	
Do	800	1.0	.5	.0	.0	

XYLENE FORMATION.

Table 40 shows the figures on xylene formation and indicates that although xylene may be formed in moderate quantity from cymene the amounts produced from toluene and benzene are exceedingly small. The formation of xylene seems to be favored by low temperatures and low pressure.

TABLE 40.—*Results of cracking four aromatic hydrocarbons under various conditions of temperature and pressure, showing percentage of xylene formed.*

Hydrocarbon cracked.	Temperature.	Percentage of xylene formed at pressure of—				Average percentage formed.
		Vacuum.	1 atmosphere.	12 atmospheres.	18 atmospheres.	
	°C.					
Cymene	650	17.8	5.9	4.0	4.9	5.4
Do	725	8.9	5.2	2.1	2.8	
Do	800	9.5	1.6	1.6	.0	
Xylene	650	80.0	37.7	31.7	45.0	30.6
Do	725	52.5	40.0	27.0	26.0	
Do	800	35.6	4.5	7.9	1.1	
Toluene	650	3.4	.0	.0	.0	.9
Do	725	4.2	.0	.7	.0	
Do	800	.0	.0	1.2	1.6	
Benzene	650	.8	.0	.0	.0	.4
Do	725	1.4	.7	.0	.0	
Do	800	1.7	.5	.0	.0	

CYMENE FORMATION.

Cymene is formed in negligible quantity from xylene, toluene, and benzene, and not at all from naphthalene and anthracene, as is shown by the results presented in Table 41, following. This compound appears to be particularly unstable under the conditions of the experiments, as is evidenced by the fact that only a small amount (an average of 12 per cent) remained unchanged when this hydrocarbon was employed as initial material.

TABLE 41.—*Results of cracking four aromatic hydrocarbons under various conditions of temperature and pressure, showing percentage of cymene formed.*

Hydrocarbon cracked.	Temperature.	Percentage of cymene formed at pressure of—				Average percentage formed.
		Vacuum.	1 atmosphere.	12 atmospheres.	18 atmospheres.	
	°*C.*					
Cymene	650	34.5	21.6	21.3	10.6	11.9
Do	725	17.0	5.4	3.0	7.5	
Do	800	14.5	1.6	5.7	.0	
Xylene	650	1.9	2.2	.7	.6	1.2
Do	725	2.4	6.0	.0	.0	
Do	800	2.3	.9	.0	1.5	
Toluene	650	.0	.5	.0	.0	.9
Do	725	.2	1.6	1.2	.9	
Do	800	.1	.0	.0	6.7	
Benzene	650	.4	1.0	.0	.0	1.2
Do	725	2.8	.7	.0	.0	
Do	800	6.9	3.0	.0	.0	

NAPHTHALENE FORMATION.

The results presented in Table 42, showing the naphthalene formed in the experiments, indicate that naphthalene is generally one of the products of the cracking of the monocyclic compounds cymene, xylene, toluene, and benzene. This is in part contrary to the observation of Haber[a], who states that although diphenyl may be produced by the pyrogenic decomposition of benzene, naphthalene is not so formed. Diphenyl was undoubtedly produced in the present experiments, although no attempt was made to isolate or identify it. Direct evidence was obtained of the formation of naphthalene and in some experiments it appeared in considerable quantity in the cooled distillates.

TABLE 42.—*Results of cracking four aromatic hydrocarbons under various conditions of temperature and pressure, showing qualitative indications of naphthalene formation.*

Furnace conditions.		Naphthalene present (+) or absent (−) as result of cracking—			
Temperature.	Pressure.	Cymene.	Xylene.	Toluene.	Benzene.
°*C.*	*Atmosphere.*				
650	(*a*)	+	−	+	+
650	1	−	−	+	+
650	12	+	−	−	+
650	18	+	+	+	+
725	(*a*)	−	−	+	+
725	1	+	+	+	−
725	12	+	−	+	+
725	18	−	+	−	+
800	(*a*)	−	−	+	+
800	1	−	−	−	+
800	12	−	−	+	+
800	18	−	−	−	+

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[a] Haber, F., Theorie der pyrogen reationen aliphatischer Kohlenwasserstoffe: Ber. Deut. chem. Gesell., Jahrg. 29, Bd. 3, 1896, p. 2691.

ANTHRACENE FORMATION.

The indications of anthracene formation in the experiments were not particularly satisfactory, but the experiments established the fact that anthracene may be formed by the heating of the other hydrocarbons. Anthracene is formed with some readiness by the cracking of naphthalene and it occasionally appears in the higher distillation cuts from the runs with liquid hydrocarbons. Table 43, showing the qualitative results of tests for anthracene formation, follows.

TABLE 43.—*Results of cracking five aromatic hydrocarbons under various conditions of temperature and pressure, showing qualitative indications of anthracene formation.*

Furnace conditions.		Anthracene present (+), or absent (−) as result of cracking—				
Temperature.	Pressure.	Cymene.	Xylene.	Toluene.	Benzene.	Naphthalene.
° C.	*Atmosphere.*					
650	(a)	−	−	−	+	−
650	1	−	−	−	−	−
650	12	−	−	−	−	−
650	18	+	−	−	−	
725	(a)	−	−	−	−	+
725	1	−	+	−	−	+
725	12	−	−	+	+	+
725	18	−	−	−	+	
800	(a)	−	+	+	−	+
800	1	−	−	−	−	+
800	12	−	−	+	−	+
800	18	−	−	−	−	

^{a} Vacuum.

EXPERIMENTS WITH PETROLEUM AS INITIAL MATERIAL.

To support the evidence obtained from cracking experiments in which pure aromatic hydrocarbons were the original material a different angle of attack was adopted. The order of formation having been demonstrated, it appeared that of a necessity similar relations should maintain among aromatic hydrocarbons derived from petroleum. An investigation was carried out in which cracked oils varying in specific gravity from 0.84 to 1.05 were produced in quantities sufficient to permit careful analysis.

The furnace employed was a tube 10 feet long and 8 inches in diameter, heated by gas. Its use permitted a feed of 15 to 20 gallons an hour of original oil and a correspondingly large recovery—20 to 50 per cent of the original.

Although the use of such a large cracking furnace had the advantage of yielding sufficiently large quantities of cracked oil to permit the making of satisfactory analyses there were the following difficulties that should receive mention, although in no way did they decrease the value of the results obtained. Pressures could be regulated as well in the large apparatus as in the small one, but the accurate control of temperature throughout the whole of a tube 10 feet long and 8 inches in diameter obviously involved difficulties. The runs were conducted with much care and with a knowledge of details of manipulation acquired in the course of several hundred previous experiments of the same sort. The temperatures recorded are, however, representative only of a moderately accurate average and no stress is laid on them in the present connection. The conditions of the runs were so regulated as to produce cracked oils of specific gravities varying over a considerable range.

GENERAL SCHEME OF PROCEDURE.

The original material was a special fuel or gas oil, a distillate obtained from Pennsylvania crude. Its specific gravity was 0.83 and its boiling range between 300° and 400° C.

Runs were made with the furnace described above, the condensed liquid (cracked oil) being the only product collected and examined.

Percentage recoveries were determined and specific gravities measured. The method of analysis was that of distillation and specific gravity.

The methods used in obtaining naphthalene and anthracene were not of the same degree of accuracy as those for obtaining the monocyclic hydrocarbons, but the results proved to be satisfactory. The two hydrocarbons mentioned were contained in the cuts above 175° C. left after the first distillation. This cut was subjected to further fractionation and two parts separated, one boiling at 175° to 270° C., and the other at 270° C. up to the point where actual tar began to come over. The quantities of the two hydrocarbons formed in these cuts were determined by the simple process of chilling, pressing out, and weighing the separate solid.

RESULTS OF EXPERIMENTS.

The results of the experiments appear in Table 44, following. The figures in the first five columns have to do with the cracked oil, its conditions of production, and its properties.

TABLE 44.—*Results of cracking a fuel-oil distillate under furnace conditions so regulated as to produce recovered oils of various specific gravities, showing percentages of benzene, toluene, xylene, naphthalene, and anthracene formed.*

[Analyses performed by the method of distillation and specific gravity; percentages are by weight.]

Furnace data.					Distillation analysis.					Percentage in cracked oils of—					Specific gravity of recovered oil.
Pressure, pounds per square inch.	Temperature, °C.	Oil feed, gallons per hour.	Oil recovered.		Percentage in cut at 175° C.	Specific gravity of cut to 175° C.	Percentage in cuts to 175° C. of—			Benzene.	Toluene.	Xylene.	Naphthalene.	Anthracene	
			Per cent.	Specific gravity.			Benzene.	Toluene.	Xylene.						
200	[a] 600	14	30.0	1.018	36.2	0.874	52.9	28.8	8.6	19.1	10.4	3.1	14.1	9.6	1.018
200	700	14	20.0	1.000	39.3	.876	52.6	28.6	11.7	20.7	11.2	4.6	8.1	3.8	1.000
250	650	15	23.5	.992	39.5	.873	50.6	30.9	11.2	20.0	12.2	4.4	7.0	.0	.992
250	700	15	22.5	.978	42.4	.870	47.6	29.3	11.9	20.2	12.4	5.0	2.0	.0	.978
250	650	15	23.5	.954	55.8	.866	29.6	31.5	19.4	16.5	17.8	10.8	.0	.0	.954
200	625	14	30.0	.938	44.8	.850	27.4	30.0	15.7	12.3	13.4	7.0	.0	.0	.938
250	600	15	31.2	.900	52.2	.817	15.1	23.0	13.6	7.9	12.0	7.1	.0	.0	.900
200	550	14	55.0	.837	39.0	.762	.0	10.2	7.1	.0	4.0	2.8	.0	.0	.837

[a] Obviously experimental error.

DISCUSSION OF TABULATED DATA.

The results shown in the table are not particularly easy to interpret. By plotting, however, the indications of importance may be brought out in a striking manner. In figure 35 curves have been plotted, the abcissas showing specific gravities of recovered oil and the ordinates percentages by weight of the five hydrocarbons contained in the oil. Average curves are given, as the irregularities, due to experimental error (imperfect condensation), would otherwise lessen the value of the indications obtained. It is to be noted also that no pretense is made of having analyzed the cracked oils for all constituents. The five hydrocarbons were chosen on account of their commercial importance and because they were typical.

It will be noted that toluene and xylene were found in a sample that yielded no benzene and that the curves for toluene and xylene bear a more or less constant relation to each other. Xylene percentages are about half those of toluene, and the maximum for each of the two curves occurs at the same point on the horizontal axis, this representing most favorable conditions for the formation of these two aromatics. Benzene is a product of more vigorous cracking; it begins to form at a higher point on the scale adopted and has a higher maximum. The proportion of benzene formed runs between the proportions of toluene and xylene until the latter two pass their maximums. The benzene maximum is, however, greater than that of toluene.

The figures covering the formation of naphthalene and of anthracene are few, but furnish important indications. Naphthalene formation begins at about the point where the formation of xylene

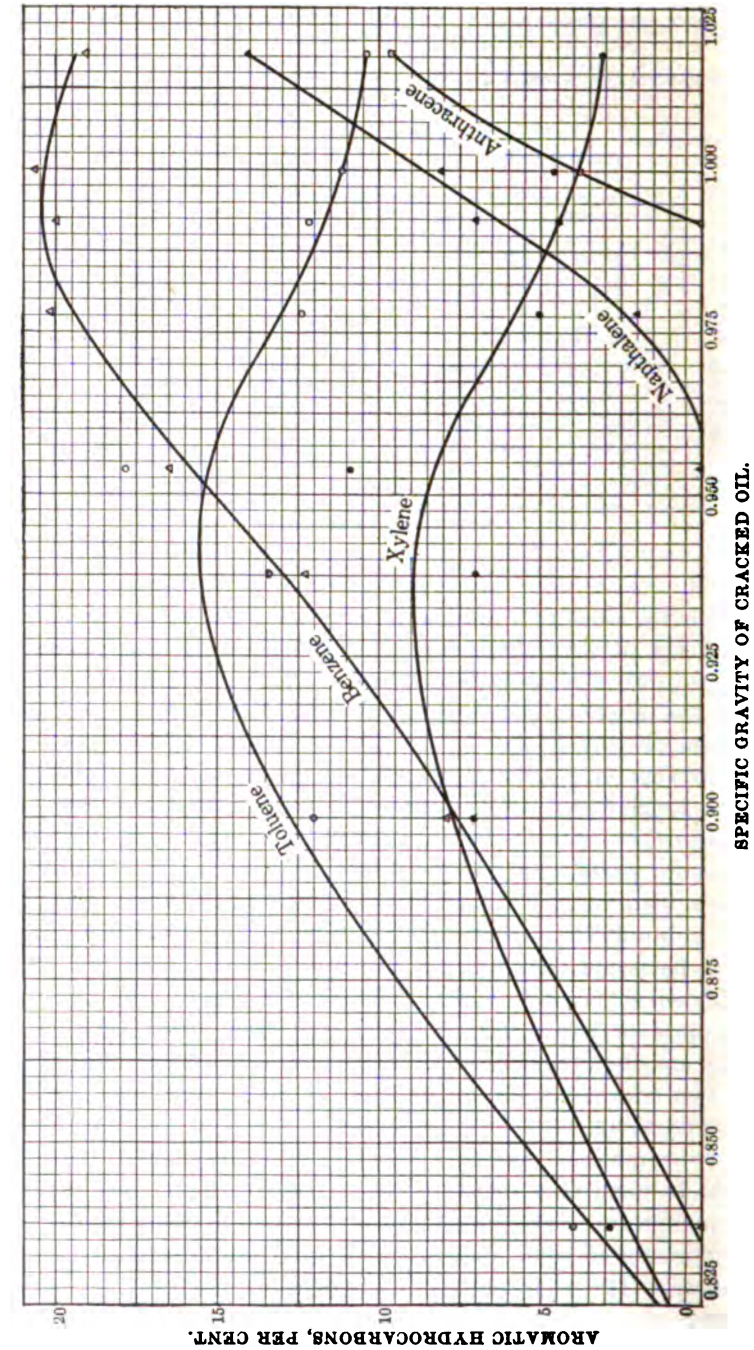


FIGURE 35.—Curves showing varying amounts of five aromatic hydrocarbons recovered in cracked oils of various specific gravities.

and toluene commences to fall off. The naphthalene curve ascends rapidly at the upper limit of the range of temperatures used in the experiments, and there is no indication that it has begun to approach its maximum. Cracking conditions to produce anthracene must be even more strenuous than those to produce naphthalene.

GENERAL SUMMARY.

The results of the experiments described show that from cymene it is possible to produce by "cracking" all the other hydrocarbons of the series, such as xylene, toluene, benzene, naphthalene, and anthracene. From xylene results toluene, benzene, naphthalene, and anthracene, but no cymene. Toluene yields benzene, naphthalene, and anthracene, but no cymene and no xylene. Benzene goes to naphthalene and anthracene, but not to any of its higher homologues. From naphthalene anthracene is readily obtainable, but none of the monocyclic hydrocarbons, benzene, toluene, xylene, and cymene, is produced. Anthracene yields no naphthalene, but goes to tarry matter, carbon, and gas.

Likewise it appears that the formation of higher benzene homologues is favored by less strenuous conditions of cracking than constitute an optimum for benzene. Polycyclic hydrocarbons are shown to be decomposition products of monocyclic hydrocarbons, as production of the latter falls off under cracking conditions that favor the formation of such typical compounds as naphthalene and anthracene.

On the basis of the evidence at hand it seems justifiable to state that the course of the cracking reaction in the aromatic series may be indicated as follows and that practically no reverse action occurs.

Higher benzene homologues $\rightleftarrows$ lower benzene homologues $\rightleftarrows$ benzene $\rightleftarrows$ (diphenyl) $\rightleftarrows$ naphthalene $\rightleftarrows$ anthracene $\rightleftarrows$ carbon and gas.

APPLICATION TO OTHER FIELDS OF PRINCIPLES ESTABLISHED BY HYDROCARBON REACTIONS.

The technical and industrial importance of the different reactions reported herein is much greater than their application to the treatment of petroleum alone would indicate. The principles involved are equally applicable to the treatment of coal, lignite, peat, bitumen, or any other substance capable of yielding liquid hydrocarbons. In fact, the field for application of these principles is even greater as regards these other substances because of their greater extent and production. The output of petroleum in the United States, although 65 per cent of that of the whole world, is decidedly secondary, on the basis of volume and tonnage, to that of solid fuels, indicating at once the tremendous possibilities in the production of tar and other hydrocarbons from coal and the other materials enumerated.

In view of the widely varying percentages of the different aromatic hydrocarbons obtainable from a crude oil, and in view of the course of reactions among aromatic hydrocarbons, a most significant feature is that there are present in the tars resulting from most present-day distillations of coal a small percentage of low-boiling aromatic hydrocarbons, such as benzene and toluene, and a very large percentage of aromatic hydrocarbons of large molecular weight such as naphthalene and anthracene. The latter undoubtedly are formed in some stage of the operation from monocyclic aromatic hydrocarbons. In practically all coal tars there is less than one part of toluene to three parts benzene. It is furthermore common information that naphthalene, methyl-napthalenes, methyl-anthracenes, anthracenes, etc., are present in large proportions. In the light of the investigations reported, which unquestionably indicate the course of reaction to be first in the formation of the higher members of the benzene series, then progressively to the formation of toluene, benzene, naphthalene, anthracene, carbon, and gas, it is to be concluded that practically every present coal-distillation process carries tho cracking to the point of ruinous decomposition of benzene and toluene in the formation of the higher boiling aromatics. The Bureau of Mines is at present investigating this feature.

PART II.—DESCRIPTION OF LARGE-SCALE DEVELOPMENT OF BENZENE-TOLUENE AND GASOLINE PROCESSES.

PURPOSE OF EXPERIMENTS.

In connection with the description of and comment on the large-scale development of the benzene-toluene process which follows, it should be kept in mind that the object in view was to demonstrate the commercial feasibility of the process. Certain refinements in mechanical equipment were out of the question at the outset for two reasons. The first was that such devices were not suited to minute study of each step in the process; the second was that they could be installed at any time after the commercial possibilities of the process had been demonstrated. For instance, such obvious improvements as feeding the oil by a single pump through a common header, or the use of one large condenser for all the tubes in place of individual condensers for each tube, were set aside in favor of less desirable mechanical arrangements by which each tube could be kept as a separate unit and its products analyzed apart from those of the remaining tubes.

As regards the condenser installations described herein, it must be borne in mind that these were not intended for continuous operation, 24 hours a day, as their capacity was limited to runs of 4 to 5 hours' duration. For short runs they served the purpose admirably and made possible the analysis and study of the products from each tube, but for long runs their efficiency was low. The recovered oil often left the condensers at the end of a run of considerable duration at a temperature higher than the boiling point of benzene and toluene. This high temperature naturally caused heavy evaporation losses and is responsible for the relatively low and inconsistent yields which are reported for the period of purely experimental development covered by this bulletin. It can safely be stated that the total yield of low-boiling aromatic hydrocarbons reported could have been doubled by the installation of an adequate condensing system supplemented by apparatus for washing or scrubbing the uncondensed gases.

It will readily be recognized that it was first necessary to prove that the process would work as well on a commercial basis as in the laboratory. This could be done only at the sacrifice of a very considerable percentage of the product formed. Elimination of this loss was regarded as a mere matter of course, to be taken up at will.

These losses continued, however, throughout the period covered by the data published in this bulletin. At the present writing a large and efficient condenser with an effective cooling surface of approximately 250 square feet is being installed, also a large gas scrubber.

Another feature for which allowance must be made in considering the discussion of mechanical apparatus is the fact that all commercial-scale equipment was designed on the basis of experience with laboratory-size apparatus. The deficiency in mechanical perfection is of less importance than the fact that it was possible on such a meager basis to complete an installation that required so few radical changes in its development and has worked so well.

No attempt has been made to conceal any weaknesses in the development of the process. Indeed emphasis has been placed on them in the belief that the negative information thus furnished is of equal if not greater value than that supplied by the relation of favorable facts only.

Too rigid deductions should be not drawn from the data included in the tables. The conditions under which the large-scale experiments were conducted, the lack of adequate condensation, which reduced the yields as much as 50 per cent below what could have been recovered, variations in rate of flow of oil into the tubes, and inaccuracy in the reading of temperatures, all had their effect, so that the accuracy in the results in nowise compares with that in the laboratory experiments covered by Part I. The important point to be observed is the fact that low-boiling aromatic hydrocarbons were formed in commercial quantities, and that these quantities can be largely increased with proper condensing equipment and other mechanical improvements.

PUBLIC DEMONSTRATION OF PROCESSES.

In February, 1915, the results which had shaped themselves from the experimental work were communicated to the Director of the Bureau of Mines. After the conclusions had been verified a public demonstration of the operation of the two processes was given in the laboratory of the department of engineering chemistry, Columbia University, New York, on March 8, 1915. Invitations had been extended to representatives of the National Petroleum Refiners' Association through its secretary, Judge C. D. Chamberlain, of Cleveland, Ohio, and to representatives of the Standard Oil Co. of Indiana, the General Chemical Co., the E. I. du Pont de Nemours Powder Co., the Aetna Explosives Co., and a number of other concerns. The demonstration was witnessed by more than 100 persons, including oil men and others interested in the industrial application of the processes.

Previous announcement of the discovery of the processes had been made by the Secretary of the Interior, under date of March 1, 1915.

Following this announcement an offer to cooperate in the development of the benzene-toluene process was submitted by the Aetna Explosives Co., of New York. Action on this offer was deferred till after the demonstration in order that all who so desired might have an opportunity to indicate their willingness to cooperate in like manner.

At the time of this demonstration further announcement was made by the Director of the Bureau of Mines that the bureau was ready to receive and consider offers of cooperation in the development of the processes, in order to demonstrate their practicability in large-scale operations and under the stress of competitive conditions.

AGREEMENT WITH AETNA EXPLOSIVES COMPANY FOR DEVELOPMENT OF BENZENE-TOLUENE PROCESS.

The proposal of the Aetna Explosives Co. to cooperate with the Bureau of Mines in demonstrating the commercial practicability of the benzene-toluene process was accepted as of March 9, 1915, and a contract entered into whereby the Aetna company pledged itself to the expenditure of not less than $200,000 in the development of the process. A copy of the contract follows:

This agreement made and entered into in triplicate this 9th day of March, A. D. 1915, by and between The United States Bureau of Mines, by its duly authorized officers, hereinafter called the first party, and The Aetna Explosives Company, Incorporated, a corporation organized and existing under the laws of the State of New York, and having its principal office at 2 Rector Street, in the city, county, and State of New York, and duly authorized to carry on business in the State of Pennsylvania, hereinafter called the second party.

Whereas the said Bureau of Mines is by the act of February 25, 1913, (37 Stat., 681) especially charged with the duty of conducting scientific and technologic investigations concerning the treatment and utilization of mineral substances with a view to increasing efficiency, economic development, and conserving resources through the prevention of waste, and

Whereas the sundry civil appropriation acts approved August 1, 1914, and March 4, 1915, respectively contain appropriations of twenty-five thousand and thirty-five thousand dollars for inquiries and investigations concerning the mining, preparation, treatment, and utilization of petroleum and natural gas, with a view to economic development and conserving resources through the prevention of waste, and

Whereas Dr. Walter F. Rittman, a chemical engineer in the employ of the first party, has recently discovered a new and valuable process for the manufacture of benzene, toluene, and other aromatic compounds from petroleum hydrocarbons (hereinafter referred to as the process), and

Whereas it is desired to develop said process for the benefit of the people of the United States and to secure the cooperation of the said first and second parties in the development of said process with a view to conserving the petroleum resources of the nation through the utilization of waste and inferior oils, and in promoting efficiency in the treatment of petroleum oils, in which work it is desirable to have the cooperation of chemists, mechanical engineers, electricians, and other employees in the employ of either or both of the aforesaid parties:

Now, therefore, this agreement witnesseth that in consideration of the cooperation of the said first and second parties in the development of the process and of services to be furnished and moneys to be expended by each of the said parties, all as hereinafter set forth, the first and second parties do hereby agree as follows:

1. The first party shall furnish the services of a competent chemical engineer in the person of Dr. Rittman or his successor and an assistant or assistants, in its discretion to cooperate in the development of the process and the designing and perfection of the machinery and other equipment necessary for the practice of the said process, and shall pay the salary and expenses of said chemical engineer. The salary and expenses of the said assistant or assistants furloughed for this purpose by the first party to be paid by the second party.

2. The second party, within fifteen days from the date hereof, shall provide, in the City of Pittsburgh, Pennsylvania, or elsewhere, a building suitable for the installation of a plant for putting the said process into practical operation, and shall provide in said building suitable and necessary laboratory facilities and accommodations and in consideration of the premises the second party agrees that the facilities of such laboratory, so far as may be done without interfering with the work incident to the development of the process, shall be available to other employees of the first party engaged on independent research work under the supervision of the aforesaid chemical engineer, the salaries and expenses of all such additional employees to be paid by the first party out of the funds appropriated for such purposes.

3. The second party agrees that it will install the process and work out its applicability on a commercial scale of magnitude and shall not desist therefrom without the consent of the first party until it shall have expended the sum of two hundred thousand dollars, or such part thereof as may be necessary to sufficiently demonstrate the commercial applicability of the process.

4. It is expressly agreed by and between the parties hereto that all patentable devices, improvements, processes, and developments originated by the employees of the first party or by the employees of the second party, working under the direction of the employees of the first party, shall at the option of the first party be patented in the United States and the patents assigned to the Secretary of the Interior, as trustee for the public, and the parties hereto shall, so far as possible, take appropriate steps to make this provision effective.

5. It is further expressly understood and agreed that the first party shall be at liberty to make public from time to time all such information concerning the development of the process, the details of plant construction, and methods of operation as it may consider of public benefit and essential to the understanding of the practical workings of the process by the general public: *Provided, however*, That no publication shall be made of any information acquired during the life of this agreement regarding the business or trade secrets of the second party other than as hereinbefore provided, and the second party hereby agrees that no information concerning the process, details of plant construction, or methods of operation shall be made public by it or by any of its employees with its knowledge or consent during the term hereof without the approval of the first party.

6. It is further understood and agreed that this cooperative work by the second party shall be under the control and supervision of the chemical engineer aforesaid or his assistant and that the installation and operation necessary for the proper commercial test of the process shall be in accordance with his direction until such time as this agreement shall have expired or have been terminated, or until such time as the first party shall consider that a sufficient demonstration has been made of the practicability of the process and further cooperation is unnecessary. Nothing herein contained shall be construed to obligate the first party to devote the entire time of the said chemical engineer, his assistant, or assistants to the work herein contemplated other than is reasonably necessary to carry out the objects of this agreement, and the first party reserves the right to utilize such portion of the time and services of the

aforesaid engineer and assistants as can be utilized without interfering with the progress of the work contemplated hereunder.

7. This agreement shall be in force and effect until June 30, 1915, or for such further length of time in no case extending beyond June 30, 1916, as may be necessary for the accomplishment of the objects hereof. Said agreement may be terminated by either party hereto upon the expiration of six months from the date hereof upon giving written notice to this effect to the other party hereto thirty days in advance of the proposed termination.

In witness whereof, on the day and year first hereinbefore written, the United States Bureau of Mines has caused these presents to be executed by Van. H. Manning, acting director, thereunto duly authorized, and on the day last aforesaid The Aetna Explosives Company, Incorporated, by resolution of the board of directors hereto attached has caused these presents to be executed by A. J. Moxham, its president, and its corporate seal to be affixed hereto by W. H. Evans, its secretary.

Witnesses:

(Signed) VAN. H. MANNING,
Acting Director, Bureau of Mines.
THE AETNA EXPLOSIVES COMPANY, INC.,
By (signed) A. J. MOXHAM,
President.
Attest:
(Signed) W. H. EVANS,
Secretary.

Approved: Mar. 11, 1915.
(Signed) FRANKLIN K. LANE,
Secretary of the Interior.

POLICY OF THE DEPARTMENT OF THE INTERIOR REGARDING USE OF GASOLINE AND BENZENE-TOLUENE PROCESSES.

Applications for patents on the gasoline and benzene-toluene processes were filed early in March, 1915, and the applications have since taken their regular course in the Patent Office.

In order that the fullest possible benefit may be realized by the public from the discoveries, it has been decided to have the patents, when granted, administered by the Secretary of the Interior as trustee who will issue licenses or permits to all those seeking to employ the processes. No royalty or other monetary payments will be required. Although the use of the processes is to be free of charge to all citizens of the United States, it has been considered necessary for the protection of the basic patents, that licensees shall assign to the Secretary of the Interior, as trustee, all patentable improvements, relating to the processes, that may be developed by them during the employment of the process. It is felt that if each licensee obtains the benefits of the improvements worked out by his colicensees there is no particular hardship in his being compelled to turn over to the Government such improvements of a patentable character as may originate with himself or his employees. The justness of this condition, it is believed, will readily be recognized.

Although, legally speaking, no license or permit can be granted for the use of a patentable invention prior to the grant of letters patent the Department of the Interior has adopted the use of a provisional form of license as a means of permitting the general use of the processes while the patents are pending. A copy of the form of license adopted is given below.

LICENSE FOR USE OF THE RITTMAN .... PROCESS—NONEXCLUSIVE—WITHOUT ROYALTY.

This agreement, made and entered into in duplicate this .... day of ...., A. D. 191.., between the Bureau of Mines, acting in this behalf by ...., Director, hereinafter called the first party, and ....
....
hereinafter called the second party:

Witnesseth, That whereas Walter F. Rittman, a chemical engineer in the employ of the first party, has discovered a new and valuable process for the manufacture of .... from heavy liquid hydrocarbons (hereinafter referred to as the process), and

Whereas the said discovery is covered by pending application for letters patent; and

Whereas, the second party is desirous of employing such process in advance of the issuance of a patent:

Now, therefore, the said first and second parties do hereby agree as follows:

1. The first party, in consideration of the covenants and agreements hereinafter contained to be observed and performed by the second party, hereby licenses and empowers the second party to use the said process, subject to the conditions hereinafter named, at .... factory in ...., and in no other places.

2. The second party agrees that all devices, improvements in processes, equipment, apparatus, or development, or any matter or thing which may be necessary or desirable to be used in connection with the said process, originated by the second party, or by its agent or employees, during the life of this agreement, shall be promptly and fully communicated to the first party in order that letters patent thereon may be applied for in the discretion of the first party, the cost of obtaining such letters patent, including the Patent Office fees, to be paid by the second party.

3. The second party further agrees to transfer and assign, or cause to be transferred and assigned, to the Secretary of the Interior, as trustee for the public, or to such other person or agent as may be designated by the first party for such purpose, all the right, title, and interest of said second party or of its agents or employees in and to the aforesaid devices, improvements, processes, equipment, apparatus, development, matters or things as regards the United States, and further agrees to assign and transfer, or cause to be transferred and assigned, all right, title, and interest as aforesaid as regards all countries or places outside of the United States, to the said Walter F. Rittman, his heirs or assigns.

4. The second party further agrees not to make public in any way any details of the process which may be communicated to it, or which it may learn by reason of the employment of such process, without the prior obtained consent of the first party.

5. The second party further agrees to furnish the first party upon request with complete working plans and specifications of plant construction, equipment, apparatus, machinery, improvements, and devices of whatsoever nature or character used in the practice of or in connection with the aforesaid process, and to furnish such party with such other pertinent information and records appertaining to the same as it may from time to time request.

6. The second party lastly agrees that the process aforesaid shall be used only in the treatment of liquid hydrocarbons produced in the United States of America: *Provided,*

That the process may be employed in the treatment of foreign-produced oils in the United States upon payment to Walter F. Rittman, his heirs or assigns, of a reasonable royalty to be mutually agreed upon between the said Rittman, his heirs or assigns, and the second party.

7. The first party shall be at liberty to make public from time to time all such information concerning the development and use of the process in the aforesaid factory of the second party, as it may consider of public benefit and essential to the working of the process by the people of the United States. No publication shall be made of any information acquired regarding the business and trade secrets of the second party other than as hereinabove provided.

8. The first party agrees to grant to the second party, upon the issuance of letters patent covering the aforesaid process, a license to employ such process in the treatment of petroleum oils or other liquid hydrocarbons produced in the United States upon the same terms and conditions as the process may be permitted to be employed by any and all other persons in the United States.

9. It is further mutually understood and agreed that the first party, in its discretion, shall have the right to terminate this agreement for the use of said process upon violation of any of the substantial terms or conditions hereof or for nonuser for a period of not exceeding six calendar months. Waiver of any specific cause of forfeiture shall not be construed to prevent forfeiture for any other or like cause occurring thereafter.

In witness whereof, on the day and year first hereinbefore written, the parties hereto have subscribed their names and affixed their seals.

....

Director, Bureau of Mines.

....

PROCEDURE TO BE FOLLOWED IN OBTAINING A LICENSE.

Application for licenses should be addressed to the Director of the Bureau of Mines, Washington, D. C. The application will receive prompt consideration and such advice along technologic lines and general assistance as can be granted will be extended. Because of the limitations put on the use of its funds by congressional enactment, and the lack of a suitable organization for the purpose, the bureau can not undertake to supervise or actively assist in the construction or operation of any plant for the employment of either or both processes. It will, however, undertake to give such general information and assistance as it can, in the light of knowledge and experience acquired in connection with the operation of the processes.

LARGE-SCALE DEVELOPMENT OF BENZENE-TOLUENE PROCESS.

Under the terms of the agreement with the Aetna Explosives Co., the contents of which have been set forth, the work incident to the development of the benzene-toluene process on a scale of commercial magnitude was begun at Pittsburgh, Pa. A plant was acquired and the experimentation was begun which was to result in the successful working out of the process on a commercial scale.

The general layout of the plant where the process has been operated is shown in Plate I, *A*.

The engineering firm of Mackintosh, Hemphill & Co., of Pittsburgh, Pa., was retained by the Aetna company for the purpose of developing the mechanical apparatus necessary for the large-scale utilization of the process. Mr. C. C. Stutz was placed by this company in direct charge of engineering matters, under the general supervision of Dr. F. L. Slocum, as representative of the Aetna interests.

EXPERIMENTS WITH VARIOUS SIZES OF TUBES.

The original apparatus which was used in the laboratory experiments, it will be recalled, consisted of a tube 1½ inches in diameter by 3 feet in length. When the development work was undertaken, a tube of the same internal diameter, but 4½ feet in length, was tried out. A small number of experiments sufficed to show that the longer tube gave the same general results.

The next step was an increase in the diameter of the tube from 1½ inches to 2½ inches, the length being 4 feet. This tube gave equally satisfactory results, and was, therefore, promptly abandoned in favor of a tube 4½ inches in diameter and 6 feet long. The use of a cracking chamber of this size was found to give results comparable with those obtained with the original small tube. Some half dozen or more runs were made at varying temperatures and rates of feed, at a pressure of 250 pounds per square inch. At a temperature of 600° C. and a rate of feed of 8 gallons per hour an oil of 0.94 specific gravity was recovered, which on distillation in the laboratory gave a benzene yield of 12 per cent and a toluene yield of 8 per cent, on the basis of recovered oil. Sufficiently favorable results were obtained with this apparatus to justify its abandonment in favor of one of still larger size.

EXPERIMENTS WITH 4½-INCH BY 10-FOOT TUBE.

In the next series of experiments the length of the tube was increased from 6 to 10 feet, the diameter remaining the same. It was found that operations at comparable temperatures and pressures with those used in the shorter tubes gave the same relative yields of aromatic hydrocarbons. The increased length, however, which exposed the gases for a longer period of time to the influence of temperature and pressure, made it possible to increase the rate of feed from 8 to 10 gallons per hour. This change illustrates one of the important means of controlling the time element in a gaseous hydrocarbon reaction.

EXPERIMENTS WITH BATTERY OF TEN 4½-INCH TUBES 10 FEET LONG.

The results obtained with the single 4½-inch tube were of such a character as to justify experiments to determine whether or not satisfactory yields could be obtained by incasing 10 tubes in a single

A. GENERAL VIEW OF PLANT AT PITTSBURGH, PA., WHERE BENZENE-TOLUENE PROCESS WAS DEVELOPED.

B. VIEW OF GROUND FLOOR OF FURNACE BUILDING, SHOWING ARRANGEMENT AND APPEARANCE OF COMPLETED FURNACES.

furnace (combustion chamber). The decision was influenced in considerable measure by the desire of the cooperating company to obtain products in large quantities as soon as possible. Accordingly, a furnace containing a battery or nest of 10 tubes, 4½ inches in diameter by 10 feet in length, was constructed.

The oil was fed into the tubes by gravity. The amount of oil passing into the top of each tube to be vaporized was regulated by a needle valve placed between the oil-supply compartment and the flange connection on the top of the tube. A great deal of experimentation was necessary in order to keep the rate of flow constant during the continuance of a run. It was necessary frequently to change the piping that led from the oil-supply tanks to the head of the tubes in order to get the proper rate of flow. The general conclusion was reached that gravity feed of heavy oil is undesirable.

The wide variations in the results obtained from the individual tubes, owing to improper control of heating, made it necessary to reconstruct the furnace and alter the methods of heating. The use of a fan blower was found to give improved results in obtaining a proper admixture of air and natural gas, which was used for heating purposes. In each tube a stirring rod, operated by a motor drive, was used for a period of some seven weeks in this furnace. This rod consisted of a pipe to which short lengths of chains were attached, the chains being so staggered as to cover the inner periphery of the tube, as a result of centrifugal action, when the rod was revolved. The use of these stirring rods for the removal of carbon from the inner walls of the tubes was not found satisfactory in tubes of this diameter. After an average of approximately four hours' operation the tubes would be choked with carbon and it was then necessary to remove the rods and clean them of deposited carbon, and also clean the interior of the tubes. Experiments proved that when stirrers were not used the small 4½-inch tubes would operate two to three hours longer before becoming choked with carbon than when the stirring rods were used. The use of the rods, therefore, was abandoned so far as this furnace was concerned, and the runs increased in length as a result.

The shaft of the stirring rod, which naturally became highly heated, afforded an excellent place for the deposition of the free carbon caused by the decomposition of the gases. The carbon deposit would gradually build up until it choked the tube and no longer permitted the free passage of the gases. More than a month was consumed in trying to adjust the heating conditions so as to duplicate the successful results obtained with the single 4½-inch by 10-foot tube. Although individual tubes would give a fair amount of conversion, other tubes gave relatively slight efficiency. The choking of the tubes after a few hours operation necessitated a shutdown of the furnace, the removal of the rods, and cleaning of the tubes, so that during a considerable part of the day the furnace was out of operation from these causes alone. Also the frequent adjustments that were necessary in order to maintain a satisfactory gravity feed and to obtain better heating control limited the time of operation.

Despite the gloomy outlook at this stage of the operations, the confidence of Mr. A. J. Moxham, president of the Aetna company, never waned. A man of lesser courage and persistency might not have continued the experiments in the face of the discouragements and difficulties which were encountered daily. The successful development of the process is in large measure due to his unwavering support.

During the course of the experiments pyrometers were used to register furnace temperatures, but with unsatisfactory results. After a number of pyrometers had been burned out, their use was abandoned and eye reading of temperatures substituted in order to obtain proper heating control. A great deal of difficulty was experienced with the proper heating of the tubes. During many of the experiments the lower part of the tubes would show a temperature of approximately 750° C., whereas in the upper part the temperature would be only 500° C., thus reversing the conditions that should exist.

An Oklahoma crude oil was used in all the experiments with the above installation, but later results with furnaces of greater capacity have shown that crude oil is not as favorable for use as is a distillate with the gasoline, and heavy bottoms or residuum, eliminated.

In these experiments the percentage of benzene obtained, on the basis of original oil, was from two to four times that of the toluene. Later experiments with the same oil have shown that the relative percentage of benzene to toluene can be in the proportion of 3 to 2. The smaller amount of toluene obtained in this course of experiments indicates that the control conditions were not correct and that the toluene formed was degraded into benzene. This fact will be readily perceived by examination of the tabulated results of the experiments in which pure toluene was used as the original material. (See p. 114).

As experimentation with this furnace continued it became increasingly evident that the smaller tubes would not give mechanically satisfactory results. While efforts were being made to obtain a relationship of operating conditions in this furnace that would result in reasonable efficiency, experiments were in progress with single tubes of larger dimensions. As the results with the larger tubes showed a great improvement over those obtained with the 4½-inch tubes, it was definitely decided to abandon the use of this furnace.

Summarizing the experience with this installation, it was proved that the 4½-inch tube is of too small diameter to give efficient results.

It was demonstrated by later experiments that to obtain maximum efficiency a tube at least double or treble this diameter should be used.

EXPERIMENTS WITH 6-INCH BY 9-FOOT TUBE.

After the 4½-inch by 10-foot tubes were discarded a tube 6 inches in diameter and 9 feet long was used. With a tube of this size a wide range of experiments was conducted to determine the proper control conditions for the increased quantities of oil that the tube would handle. It was found that the feed could be increased to 14 gallons an hour, an increase in capacity of 4 gallons per hour over the 4½-inch tube.

An average of five runs, which were made at a temperature of 600° C. and 250 pounds pressure, and a rate of flow of 15 gallons per hour, gave 8.3 per cent toluene and 13.5 per cent benzene, on the basis of recovered oil.

Approximately 40 runs were made at temperatures ranging from 500° C. to 725° C. and at a constant pressure of 250 pounds, but with varying rates of oil feed. It was found that with Oklahoma crude oil the optimum conditions were approximately 600° C., a feed of 14 gallons, and a pressure of 250 pounds per square inch.

A smaller number of runs were made at temperatures between 600° and 725° C. and 125 pounds pressure, but the yields were considerably decreased, this result being due as much to failure to obtain the proper rate of oil feed as to the unsuitability of the pressure for converting oil to aromatic hydrocarbons.

The necessity of obtaining concrete results with a minimum loss of time made it necessary to suspend experiments with varying rates of pressure for the time being.

This tube was abandoned in favor of a tube of the same diameter, but 1 foot longer, with a view to greater capacity. It had previously been demonstrated in experiments with the smaller tubes that an increase in length permitted more oil to be fed in during a given period of time, owing to the increase in heating surface.

EXPERIMENTS WITH 6-INCH BY 10-FOOT TUBE.

With the installation of the 6-inch by 10-foot tube a force-feed pump was employed for the first time, with the object of obtaining a more constant flow than could be obtained by gravity feed, where the pressure exerted by the column of oil was relied on to overcome the pressure of the gases and force the oil into the tube. The variation in rate of feed which characterized the gravity system was eliminated to a large extent. It was found possible with the more uniform rate of flow to obtain a better adjustment of temperature and pressure conditions with relation to the amount of oil supplied.

TESTS WITH OKLAHOMA CRUDE OIL.

A series of experiments was conducted with this tube at varying temperatures and pressures in order to determine the most efficient conditions. Oklahoma crude oil was used in most of these experiments, the results of which are set forth in Table 45. Little uniformity is shown in the tabulated results unless the data be compared for the same rate of feed at different temperatures and pressures.

It will be noted that at a constant temperature and rate of feed, but with varying pressures, the percentage of benzene and toluene formed increased with increase of pressure. For example, at a temperature of 600° C. and a rate of feed of 12 gallons per hour, 4.3 per cent of benzene was formed at a pressure of 100 pounds. At a pressure of 150 pounds 4.9 per cent was formed and at a pressure of 225 pounds, 6.7 per cent. Similar variations were obtained when the pressure was increased at higher temperatures. For example, at a temperature of 750° C. and a feed of 14 gallons per hour 4.4 per cent of benzene and toluene was formed at a pressure of 175 pounds and 5.2 per cent at 225 pounds.

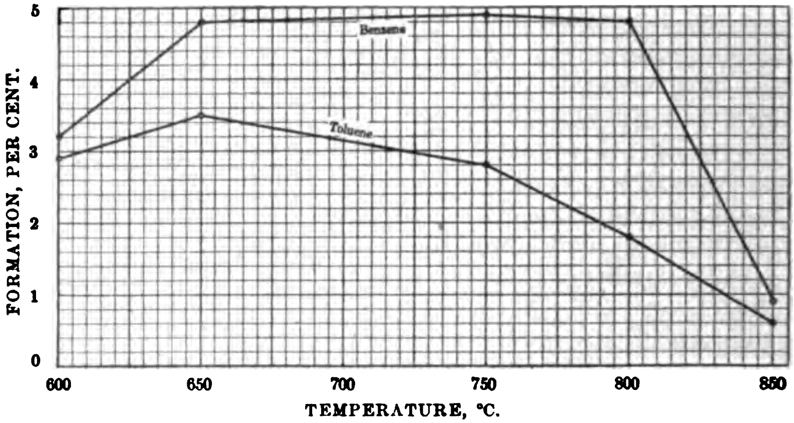


FIGURE 36.—Curves showing effect of varying temperatures on formation of benzene and toluene when original oil is cracked in 6-inch by 10-foot tube, with constant rate of feed of 17 gallons per hour, and at a constant pressure of 250 pounds per square inch. (Results low due to insufficient condensation.)

The higher temperatures, as has been stated in connection with the discussion of the course of reaction among the aromatic hydrocarbons (see p. 123), are favorable for benzene formation, but prejudicial to toluene formation. This is confirmed by the data obtained in this series of experiments. If figure 36 is examined, it will be seen that the amount of benzene formed increases progressively with increase of temperature to a temperature of 750° C., and shows a slight decrease from this point up to 800° C. As the temperature is increased beyond 800° C. the percentage of benzene formed sharply drops until at 850° C. it is practically negligible. Toluene formation, on the other hand, shows a consistent and steady decrease above a temperature of 650° C. This result again is in accord with the evidence obtained from the experiments in the small electrically heated furnace.

It will be noted that the total yields of benzene and toluene shown in Table 45 are considerably below the total percentage of aromatic hydrocarbons formed in the small laboratory apparatus. This is due to two causes; first, the percentage of xylene formed is left out of consideration, and second, and more important, the condensation was wholly inadequate to permit a full recovery of the aromatic hydrocarbons formed. The gases, after passing through the condenser, issued from the end of the pipe in the form of a white cloud, whereas the gases could not have been noted if none but fixed gases escaped. A large proportion of the volatile liquids formed were thus carried away by the gases, the quantity of which could not be determined with the facilities at hand. The percentage of loss was not of so much moment in these experiments as was the question whether the conversion could be properly carried out in the larger tubes. The object of the experiments was mainly to determine the effect of using a larger and longer pipe on the reaction products, rather than to ascertain the maximum percentage which could be recovered from the oil. It was readily recognized that efficient condensation would make a great improvement in the results. Table 45 follows:

TABLE 45.—*Benzene and toluene formed from cracking Oklahoma crude oil in a single-tube furnace (tube 6 inches in diameter and 10 feet long), at varying temperatures, pressures, and rates of oil feed.*[a]

Number of runs.	Temperature.[b]	Pressure per square inch.	Rate of oil feed per hour.	Oil recovered.	Benzene in recovered oil.	Toluene in recovered oil.	Total benzene and toluene in recovered oil.	Benzene formed on basis of original oil.	Toluene formed on basis of original oil.	Total benzene and toluene formed on basis of original oil.
	° C.	*Pounds.*	*Gallons.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
1	600	100	12	25	7.2	10.0	17.2	1.8	2.5	4.3
1	650	100	12	21	16.7	12.0	28.7	3.5	2.5	6.0
1	700	100	18	....	....	....	....	0.9	1.3	2.2
1	700	100	5	17	19.4	11.8	31.2	3.3	2.0	5.3
1	600	150	12	25	9.6	10.0	19.6	2.4	2.5	4.9
1	650	175	9	42	7.6	7.6	15.2	3.2	3.2	6.4
1	700	175	18	22	12.3	10.5	22.8	2.7	2.3	5.0
1	750	175	14	21	10.5	10.5	21.0	2.2	2.2	4.4
1	650	200	15	22	21.0	11.0	32.0	4.6	2.4	7.0
1	675	200	27	54	3.0	5.2	8.2	1.6	2.8	4.4
1	700	200	12	20	12.5	9.5	22.0	2.5	1.9	4.4
1	600	225	12	18	23.3	13.9	37.2	4.2	2.5	6.7
1	650	225	18	22	20.4	13.7	34.1	4.5	3.0	7.5
1	700	225	18	22	13.2	12.3	25.5	2.9	2.7	5.6
1	750	225	14	20	16.5	9.5	26.0	3.3	1.9	5.2
1	650	250	19	17	21.8	12.4	34.2	3.7	2.1	5.8
4	650	250	18	35	10.6	9.7	20.3	3.7	3.4	7.1
1	650	250	19	44	5.5	9.1	14.6	2.4	4.0	6.4
1	675	250	16	22	10.9	10.9	21.8	2.4	2.4	4.8
3	700	250	7	24	14.2	11.2	25.4	3.4	2.7	6.1
1	750	250	7	17	20.0	12.9	32.9	3.4	2.2	5.6

[a] Average results low, due to insufficient condensation.
[b] Temperatures measured by pyrometer attached to wall of tube.

be so adapted as to give larger quantities. Experimental evidence showing the course of the aromatic-hydrocarbon reactions, which has been presented in another part of this report, brings out this fact clearly.

This factor of control constitutes one of the greatest recommendations in favor of these processes. Absolute command over the operating conditions is made possible by causing the reaction to occur in the gaseous phase. The relation of the apparatus to the general character of the reactions is such that it is possible to predetermine the yields of the various hydrocarbons to be produced, and to vary conditions so as to increase or decrease the quantity of any one of these hydrocarbons.

CONCLUSIONS AS TO 6-INCH BY 10-FOOT TUBE.

The general results obtained with the 6-inch by 10-foot tube were satisfactory and in harmony with those obtained with other tubes. It was proved that the reaction could be carried on as successfully in a tube of this size as in the much smaller tubes which were first used. It was evident that the maximum size of furnace body had not yet been found, which suggested the advisability of experimenting with a tube of larger capacity.

EXPERIMENTS WITH 8-INCH BY 10-FOOT TUBE.

The next series of experiments were conducted with a tube 8 inches in diameter and 10 feet long. It was found possible to increase the rate of feed 5 gallons per hour over that for the 6-inch tube.

In connection with the various increases in size of tube, it will be realized that each enlargement required the preparation of new drawings, the casting of new fittings, and changes in the furnace construction. A new furnace body had to be constructed for the 6-inch tube and still another furnace body for the 8-inch tube.

No difficulty was experienced in any of these single-tube experiments with the removal of carbon from the interior of the tubes. The mechanical stirring device, which was continuously operated throughout the run, proved entirely satisfactory so that the only question to be considered was the practical one of the relative amount of carbon deposition and its bearing on operating costs.

DIFFICULTY OF ATTAINING HIGH DEGREE OF ACCURACY IN SINGLE-TUBE EXPERIMENTS.

All of the single-tube experiments were conducted in the rear of Mackintosh, Hemphill & Co.'s plant in Pittsburgh in order that their engineers, who were charged with the duty of developing the apparatus for the development company, might have the opportunity of studying the results and profiting thereby in the preparation of plans for the larger installation. A limited space was available. The recoveries were determined by bucket measurement as the condensers were tapped, and the yields calculated on the ground by means of specific gravity determinations and primary distillations. Under these conditions it was to be expected that the yields would invariably be low. Such yields, therefore, are no index of the maximum results which can be obtained under better operating conditions. This is no reflection on those connected with the development work, as general indications were sought bearing on the maximum size of apparatus that could be installed, rather than high accuracy in determining the results of single runs or experiments. Even under such conditions of operation the use of single tubes furnished unquestionably definite information. The general crudity of the arrangement can be best appreciated, perhaps, by an examination of Plate II, *A*, which shows the initial construction of the 6-inch by 9-foot tube furnace.

EFFECT OF LARGER TUBE ON OPTIMUM FURNACE TEMPERATURES.

A characteristic result of increasing the diameter and length of the tube seems to be that higher temperatures are desirable in order to obtain maximum conversion. For example, with the 4½-inch tube a temperature of 600° C. was found to give the most efficient results; in the 6-inch tube a temperature of 675° C. was found to be the optimum, whereas with the 8-inch tube a temperature of 750° C. proved to be the most satisfactory. This progressive increase in optimum temperature with enlarged periphery does not by any means indicate that tubes of greatly increased diameter can not be employed satisfactorily because of difficulty in attaining the temperature limits which the increase in diameter would make desirable. By operating at a lower temperature than that which is theoretically desirable, but with a slower rate of feed, the same results can be obtained as at the higher temperatures. At the same time the advantages in consumption capacity incident to a large cracking chamber can be gained.

COMMENT ON EXPERIMENTAL DATA FOR 8-INCH BY 10-FOOT TUBE.

TESTS WITH OKLAHOMA CRUDE OIL.

Data for the Oklahoma crude oil used are given in Table 48. It will be observed that a uniform series of runs at fixed temperatures, pressures, and rates of feed was not attempted, but that each one of these variables was changed in relation with the others so that a number of combinations could be studied. The purpose in view was to determine optimum operating conditions for the large tube used. The indications from the data are that with this tube a temperature of 675° to 700° C. and a feed of 12 to 13 gallons per hour give the best results at the operating pressure of 250 pounds per square inch.

The characteristic effect of high temperatures on the relative amounts of benzene and toluene formed, which was indicated in pages 123 and 125, is observable in the results of these tests. The percentage of benzene tends to increase progressively as the temperature is raised, whereas the relative percentage of toluene drops, a fact which clearly indicates the conversion of toluene into benzene.

It will be further noted that at a given temperature and pressure, say, for example, 700° C. and 250 pounds per square inch, the yields of benzene and toluene decrease as the rate of feed is increased. This decrease is the result of too short an exposure to heat. Experiments, which are not recorded, were conducted in which the oil was fed at rates varying from 36 to 50 gallons per hour at temperatures ranging from 575° to 650° C. which gave little or no conversion into aromatic hydrocarbons, indicating that sufficient time was not allowed for the molecular rearrangement incident to the formation of such hydrocarbons.

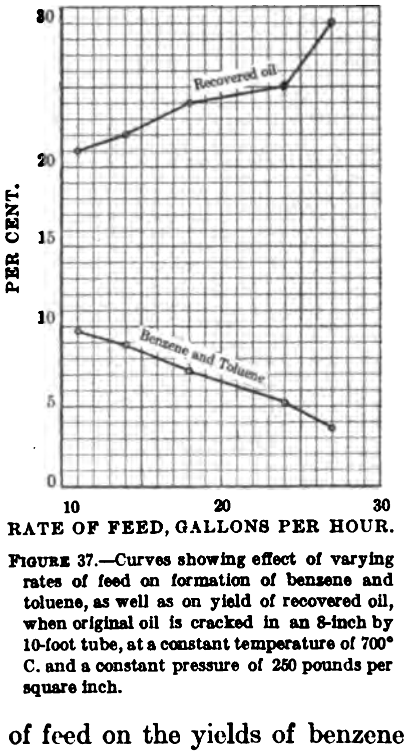
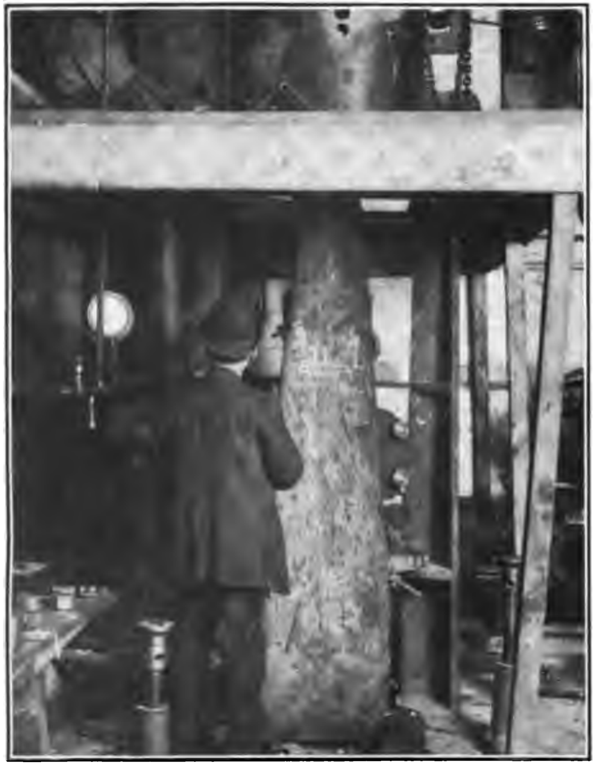
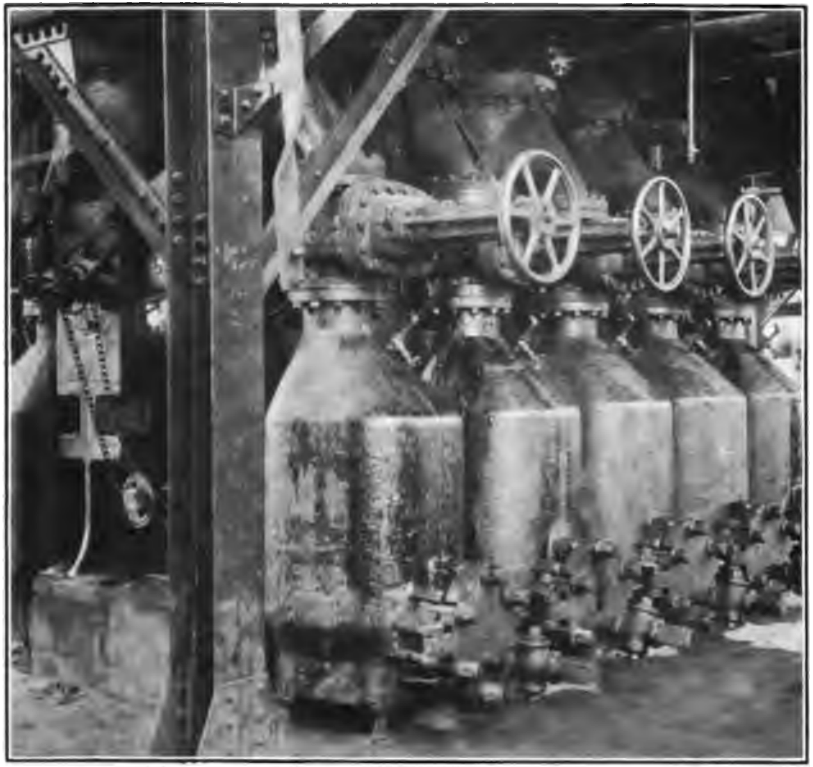


FIGURE 37.—Curves showing effect of varying rates of feed on formation of benzene and toluene, as well as on yield of recovered oil, when original oil is cracked in an 8-inch by 10-foot tube, at a constant temperature of 700° C. and a constant pressure of 250 pounds per square inch.

The effect of increasing the rate of feed on the yields of benzene and toluene is clearly indicated by figure 37, in which a constant and uniform drop in yield is shown as the rate of feed is increased. The yield of recovered oil, it will be noted, constantly becomes greater as the rate of feed increases, but is not as great, however, as is the decrease in the percentage of aromatic hydrocarbons formed. This increase in the amount of recovered oil is to be expected, as the smaller the amount of aromatic hydrocarbons formed the less is the decomposition that takes place.



A. CHARACTER OF SMALL SINGLE-TUBE FURNACE. CONDENSER IS SHOWN IN LEFT FOREGROUND.



B. ENLARGED CARBON POTS OF NEW TYPE.

Table 48.—*Benzene and toluene formed from cracking Oklahoma crude oil in a single-tube furnace (tube 8 inches in diameter and 10 feet long) at a constant pressure of 250 pounds per square inch with varying temperatures and rates of oil feed.*[a]

[For distillation analysis of Oklahoma crude oil used, see Table 29.]

Number of runs.	Temperature.	Rate of oil feed per hour.	Oil recovered.	Benzene in recovered oil.	Toluene in recovered oil.	Total benzene and toluene in recovered oil.	Benzene formed on basis of original oil.	Toluene formed on basis of original oil.	Total benzene and toluene formed on basis of original oil.
	° C.	*Gallons.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
1	650	18	27	8.5	8.9	17.4	2.3	2.4	4.7
1	650	28	25	13.6	12.4	26.0	3.4	3.1	6.5
1	675	11	23	21.0	13.5	34.5	4.8	3.1	7.9
1	675	14	25	24.0	13.6	37.6	6.0	3.4	9.4
2	675	36	75	1.8	3.6	5.4	1.3	2.7	4.0
1	700	11	21	18.7	13.8	32.5	3.9	2.9	6.8
1	700	14	25	23.2	12.0	35.2	5.8	3.0	8.8
3	700	18	22	20.0	12.7	32.7	4.4	2.8	7.2
1	700	24	25	10.8	10.0	20.8	2.7	2.5	5.2
2	700	27	29	....	....	....	1.5	2.2	3.7
2	750	17	35	9.2	10.3	19.5	3.2	3.6	6.8

[a] Results low, due to insufficient condensation.

The results of a series of runs with Oklahoma crude oil, at a constant rate of feed of 22 gallons per hour and at a constant pressure of 250 pounds per square inch but varying temperatures, are presented in Table 49. There is a constantly increasing yield of both benzene and toluene up to a temperature of approximately 650° C., the ratio of increase being nearly uniform. At higher temperatures the percentage of toluene steadily diminishes, whereas the percentage of benzene sharply increases with temperature rise up to about 750° C. Here again, however, the percentage of increase in benzene is greater at the higher temperatures than the diminution in toluene. Only

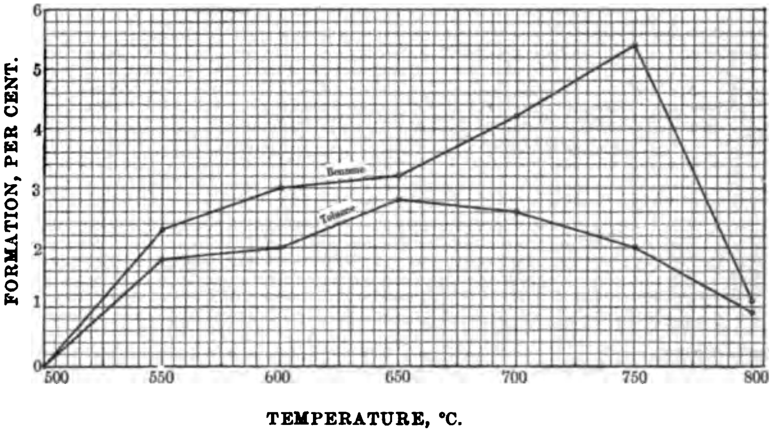


FIGURE 38.—Curves showing effect of varying temperatures on formation of benzene and toluene when an Oklahoma crude oil is cracked in 8-inch by 10-foot tube at a constant rate of feed of 22 gallons per hour and a constant pressure of 250 pounds per square inch. Average results low, from insufficient condensation.

small percentages of benzene and toluene were recovered at 800° C., indicating that at this and higher temperatures there is too much formation of polycyclic aromatic compounds and too much decomposition into the ultimate end products—hydrogen and carbon. This assumption is borne out by the rapid decrease in yield of recovered oil. These facts are shown by figure 38.

In connection with this table it will be observed that as the gravity of the recovered oil increases the combined yields of benzene and toluene approach the maximum and then decrease. This result bears out the statement previously made that the most desirable gravity of the recovered oil is one between 0.90 and 0.96. Conditions yielding oil of a gravity of 1.02 and greater are distinctly unfavorable for the formation of the lower boiling aromatic hydrocarbons, although they will give increasing yields of the heavier compounds of the aromatic series. It will be noted also that in the 8-inch tube with the rate of feed indicated conversion seems to take place at higher temperatures, a temperature of 750° C. giving the most favorable results. Throughout the work one is constantly confronted with the relation of the temperature and rate of flow. Table 49 follows:

TABLE 49.—*Benzene and toluene formed from cracking Oklahoma crude oil in a single-tube furnace (tube 8 inches in diameter and 10 feet long) at a constant pressure of 250 pounds per square inch and a constant rate of oil feed of 22 gallons per hour, with varying temperatures.*[a]

[For distillation analysis of Oklahoma crude oil used see Table 45.]

Number of runs.	Temperature.	Oil recovered.	Specific gravity of recovered oil.	Benzene in recovered oil.	Toluene in recovered oil.	Total benzene and toluene in recovered oil.	Benzene formed on basis of original oil.	Toluene formed on basis of original oil.	Total benzene and toluene formed on basis of original oil.
	°C.	*Per cent.*		*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
2	550	50	0.88	4.6	3.6	8.2	2.3	1.8	4.1
3	600	35	.92	8.6	5.7	14.3	3.0	2.0	5.0
2	650	31	.93	10.3	9.0	19.3	3.2	2.8	6.0
2	700	26	.98	16.1	10.0	26.1	4.2	2.6	6.8
2	750	21	1.00	25.7	9.5	35.2	5.4	2.0	7.4
1	800	17	1.03	6.5	5.3	11.8	1.1	.9	2.0

[a] Average results low due to insufficient condensation.

TESTS WITH DISTILLATES.

The results obtained from a series of experiments with a distillate oil are set forth in Table 50.

TABLE 50.—*Benzene and toluene formed from cracking a distillate oil in a single-tube furnace (tube 8 inches in diameter and 10 feet long) at varying pressures, temperatures, and rates of oil feed.*[a]

Number of runs.	Temperature.	Pressure per square inch.	Rate of oil feed per hour.	Oil recovered.	Specific gravity of recovered. oil.	Benzene in recovered oil.	Toluene in recovered oil.	Total benzene and toluene in recovered. oil.	Benzene formed on basis of original oil.	Toluene formed on basis of original oil.	Total benzene and toluene formed on basis of original oil.
	° C.	*Pounds.*	*Gallons.*	*Per ct.*		*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
1	600	150	14	36.3	.890	7.0	10.8	17.7	2.5	3.9	6.4
1	625	175	14	30.0	.936	14.3	13.9	28.2	4.3	4.2	8.5
1	700	200	14	20.0	1.000	23.6	13.0	36.6	4.7	2.6	7.3
2	650	200	15	24.0	.981	20.4	16.4	36.8	4.9	3.9	8.8
1	625	200	14	30.0	.938	14.7	15.3	30.0	4.4	4.6	9.0
1	600	200	14	30.0	.873	8.0	11.4	19.4	2.4	3.4	5.8
1	550	200	14	55.0	.837	.0	4.2	4.2	.0	2.2	2.2
2	700	250	15	22.5	.998	22.4	12.9	35.3	5.0	3.9	8.9
2	650	250	15	23.5	.972	20.2	15.7	35.9	4.8	3.7	8.5
1	600	250	15	31.2	.900	9.1	12.8	21.9	2.8	4.0	6.8

[a] Average results low, due to insufficient condensation.

DISTILLATION ANALYSIS OF OIL USED.

[First drop at 170° C.; specific gravity, 0.817.]

Temperature of cut.	Distillation products.		
	Volume.	Specific gravity.	Remarks.
° C.	*Per cent.*		
200	2.0	....	
250	7.3	0.792	Stuck at 290 to 296° C.
300	57.0	.818	
350	28.0	.827	
Residue	5.3	....	

Possibly the most striking feature of the results in Table 50 is the indicated ability to change the yields of either benzene or toluene by varying the temperature and pressure. The lower pressures for a given rate of feed and constant temperature are again shown to be more favorable for toluene production than the higher pressures. It appears that a moderate temperature and a moderate pressure are best suited for the maximum yields of toluene, whereas the higher pressures are most suitable for maximum yields of benzene.

The oil used in the experiments was the same as that employed in the large ten-tube furnaces with tubes 8 inches in diameter by 11½ feet in length (see Table 63). It is characteristic of this as well as all the other distillates used that the percentage formation of toluene is greater than that of benzene. On the basis of yields it is evident that this oil is more favorable for cracking to form benzene and toluene than is the Oklahoma crude oil.

The results of experiments with a distillate of a different type are presented in Table 51. Eighty-five per cent of this oil distilled over at temperatures between 300° and 350° C., as compared with 28 per cent of the other and lighter distillate. Increased yields of toluene are to be noted, as in Table 50. The average results were slightly better than with the first distillate, as the mean of the total benzene and toluene formation is 7.7 per cent as against 7 per cent. This is due to the fact that the runs were made at higher pressures. If the results for a temperature of 700° C. and a pressure of 250 pounds per square inch are compared with those in Table 50 it will be noted that the yields for the first distillate are distinctly more favorable, as 2 per cent more benzene and toluene was obtained. Consideration of the distillation cuts of the original oils will show the widely different nature of these oils.

TABLE 51.—*Benzene and toluene formed from cracking a distillate oil in a single-tube furnace (tube 8 inches in diameter and 10 feet long), at varying pressures, temperatures, and rates of oil feed.*[a]

Pressure per square inch.	Temperature.	Rate of oil feed per hour.	Oil recovered.	Specific gravity of oil recovered.	Benzene in recovered oil.	Toluene in recovered oil.	Total benzene and toluene in recovered oil.	Benzene formed on basis of original oil.	Toluene formed on basis of original oil.	Total benzene and toluene formed on basis of original oil.
Pounds.	°*C.*	*Gallons.*	*Per ct.*		*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
250	650	18	33.0	0.96	13.8	14.3	28.1	4.5	4.7	9.2
250	700	17	31.2	.95	14.0	14.5	28.5	4.4	4.5	8.9
250	700	15	30.0	.91	10.8	12.4	23.2	3.2	3.7	6.9
250	700	12	25.0	.97	13.0	13.4	26.4	3.3	3.4	7.7
250	725	22	31.2	.95	10.4	12.0	22.4	3.3	3.7	7.0
275	675	16	37.5	.91	8.0	10.0	18.0	3.0	3.8	6.8
300	675	18	40.0	.89	8.5	9.9	18.4	3.4	4.0	7.4

[a] Yields low, due to insufficient condensation.

DISTILLATION ANALYSIS OF OIL USED.

[Specific gravity, 0.859.]

Temperature of cut.	Distillation products.	
	Volume.	Specific gravity.
°*C.*	*Per cent.*	
300	10.0	0.852
350	85.2	.859
360	2.7	.867

TESTS WITH A FUEL OIL.

In addition to the two distillate oils a fuel oil was used in a series of runs made at varying temperatures and pressures. The necessity of proceeding with the experiments in a tube of larger size, in order to obtain greater capacity, prevented enough runs being made to ascertain the proper control conditions for this oil. The results for this oil, shown in Table 52, are low as compared with the two distillate oils. This does not necessarily indicate, however, that the oil is less suitable for cracking. The table follows:

TABLE 52.—*Benzene and toluene formed from cracking a fuel oil in a single-tube furnace (tube 8 inches in diameter and 10 feet long) at varying pressures, temperatures, and rates of oil feed.*[a]

[For distillation analysis of oil used see Table 45.]

Temperature.[b]	Pressure per square inch.	Rate of oil feed per hour.	Oil recovered.	Benzene in recovered oil.	Toluene in recovered oil.	Total benzene and toluene in recovered oil.	Benzene on basis of original oil.	Toluene on basis of original oil.	Total benzene and toluene on basis of original oil.
°C.	*Pounds.*	*Gallons.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
575	150	18	86	7.2	9.5	16.7	2.6	3.4	6.0
650	150	8½	25	13.6	15 2	28.8	3.4	3.8	7.2
550	200	6	83	6.1	8.5	14.6	2.0	2.8	4.8
650	250	12	28	7.2	9.7	16.9	2.0	2.7	4.7
700	250	11	28	11.4	12.9	24.3	3.2	3.6	6.8

[a] Figures reported are averages of a number of runs at the temperatures, pressures, and rates of feed shown; average results low due to insufficient condensation.

[b] Temperature measured by pyrometer attached to wall of tube.

EXPERIMENTS WITH 8-INCH BY 14-FOOT TUBE.

With a view to obtaining increased capacity, the 8-inch tube used in the previous experiments was replaced by one 14 feet long. The increased heating surface afforded by the gain in length made it necessary to determine experimentally the most favorable conditions for carrying out the reaction in this tube. Accordingly a large number of experiments were performed covering the entire range of possible temperatures and pressures, and with rates of oil feed varying from 10 to 50 gallons per hour.

It was found that the rate of feed could be increased from 22 to 30 gallons per hour with a satisfactory amount of conversion. The chemical reaction could be obtained with the same facility as in the other tubes, but the added length of heating zone caused an increase in mechanical difficulties.

It was soon made manifest that the heating control had been rendered more difficult. The center of the tube was often at white heat while both ends were relatively cool, a condition due in part to the construction of the furnace, which had been designed for the shorter tubes. The use of a tube 14 feet long caused the gas burners to be somewhat out of place for obtaining the best results. The nonuniformity in furnace heating was evidenced by the greater quantity of carbon formed. For the first time, also, the tubes displayed a strong tendency to soften in the furnace. This was due, of course, to local excess temperatures. Adding 4 feet to the length of the stirring rod also introduced new difficulties. The greater length and more intense heats, arising from inability to control the furnace temperatures, caused the rod to soften and at times buckle, a difficulty which had not been previously experienced.

Despite the fact that the reaction took place readily, the greater mechanical difficulties seemed to indicate that the optimum length of tube had been exceeded.

TESTS WITH DISTILLATE OIL.

The figures given in Table 53 include the results of experiments at widely varying temperatures and rates of feed with the same distillate oil used in the runs reported in Tables 50 and 52.

TABLE 53.—*Benzene and toluene formed from cracking a distillate oil in a single-tube furnace (tube 8 inches in diameter and 14 feet long) at a constant pressure of 250 pounds per square inch, with varying temperatures and rates of oil feed.*[a]

[For distillation analysis of oil used, see Table 50.]

Temperature.	Rate of oil feed per hour.	Oil recovered.	Benzene in recovered oil.	Toluene in recovered oil.	Total benzene and toluene in recovered oil.	Benzene formed on basis of original oil.	Toluene formed on basis of original oil.	Total benzene and toluene formed on basis of original oil.
°*C.*	*Gallons.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
650	26	44	4.3	5.5	9.8	1.9	2.4	4.3
750	38	20	14.5	16.0	30.5	2.9	3.6	6.5
800	18	20	13.0	14.5	27.5	2.6	2.9	5.5
800	25	15	16.7	16.7	33.4	2.5	2.5	5.0
800	25	25	8.0	10.0	18.0	2.0	2.5	4.5
800	35	[b] [18]	10.5	11.7	22.2	1.9	2.1	4.0

[a] Results low, due to insufficient condensation.

[b] Obviously an experimental error.

In considering the data embodied in Table 53, it should be remembered that the temperatures indicated are only approximate. Various mechanical circumstances had made it necessary to discard the pyrometers, which in the earlier furnaces were welded to the surface of the tube. In the 14-foot tube pyrometers were placed in the annular inclosures of fire brick around each tube, and were checked by optical indications. It appears probable that in a number of instances the temperatures are recorded higher than they actually were, according to the indications given by properties of recovered oils.

The low yields of benzene and toluene, as compared with those from the 10-foot tube, are to be explained upon the ground that the desired products were actually made, but after being made were decomposed into naphthalene and anthracene and the end products, hydrogen and carbon. On account of wide variations in rates of feed, results of uniform character were not obtained, and therefore satisfactory comparisons can not be made.

It is evident, however, from a study of the table, that a temperature of between 650° C. and 800° C. and a rate of feed in the neighborhood of 26 gallons per hour will give the best results, both with regard to percentage of recovered oil and value of the by-products of the reaction.

TESTS WITH OKLAHOMA CRUDE OIL.

A series of runs consisting of thirty-odd experiments with Oklahoma crude oil was made at a uniform pressure of 250 pounds and a constant rate of feed of 30 gallons per hour. Data for this series of experiments are set forth in Table 54.

TABLE 54.—*Benzene and toluene formed from cracking Oklahoma crude oil in a single-tube furnace (tube 8 inches in diameter and 14 feet long), at a constant pressure of 250 pounds per square inch, with varying temperatures and rates of oil feed.*[a]

[For distillation analysis of oil used see Table 45.]

Number of runs.	Temperature.[b]	Rate of oil feed per hour.	Oil recovered.	Benzene in recovered oil.	Toluene in recovered oil.	Total benzene and toluene in recovered oil.	Benzene formed on basis of original oil.	Toluene formed on basis of original oil.	Total benzene and toluene formed on basis of original oil.
	° C.	*Gallons.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
1	625	32	25	20.23	12.57	32.80	5.10	3.10	8.20
1	650	24	14	31.17	15.51	46.68	4.30	2.20	6.50
2	650	27	22	26.40	12.70	39.10	5.70	2.80	8.50
1	650	28	25	24.10	12.00	36.10	6.00	3.00	9.00
1	650	29	29	12.42	13.26	25.68	3.60	3.90	7.50
2	650	31	23.2	18.45	13.17	31.62	4.30	3.00	7.30
2	650	32	29	10.64	9.97	20.61	3.00	2.90	5.90
1	675	29	16	27.47	12.88	40.35	4.50	2.10	6.60
1	775	33	40	4.84	6.37	11.21	1.90	2.50	4.40
2	800	30	16.5	18.30	21.95	40.25	3.00	3.60	6.60
1	800	33	27	8.80	9.04	17.89	2.37	2.46	4.83
4	800	35	25.5	13.44	10.10	23.54	3.43	2.57	6.00
1	825	23	17	26.00	10.10	36.10	4.43	1.72	6.15
1	825	35	33	7.60	8.60	16.20	2.51	2.82	5.33
2	825	38	28.5	11.60	8.00	19.60	3.30	2.28	5.58
1	850	21	18	20.20	8.04	28.24	1.62	6.43	8.05
1	850	33	23	10.70	10.30	21.00	2.46	2.37	4.83
1	850	36	20	16.20	10.80	26.50	3.24	2.06	5.30
1	875	33	25	10.90	10.40	21.30	2.73	2.61	5.34

[a] Results low, due to insufficient condensation.

[b] Temperature readings approximate only, judged by color of tube and by pyrometer in checkerwork of furnace.

As was the case with the distillate oil the average results are less favorable than in the 10-foot tube of the same diameter. The longer exposure to heat permitted by the 4-foot increase in length evidently decomposed the low-boiling aromatics into hydrocarbons of greater molecular weight, as well as end-product gases and carbon. The results show an improvement, however, over those obtained with the same oil in the 6-inch by 10-foot tube. In that furnace the mean production of benzene and toluene was 5.7 per cent as against 6.4 in the new furnace. In considering the tabulated figures, it should be remembered that the temperatures indicated aré unquestionably high. This was the first single-tube furnace operated without a pyrometer welded to the tube, and the eye of the recorder evidently had not become sufficiently well trained to approximate temperatures accurately.

EXPERIMENTS WITH TUBE 8 INCHES IN DIAMETER AND 7 FEET LONG.

The fact that in the experiments just described the optimum length of tube had been exceeded led to experiments with an 8-inch tube 7 feet long. A number of tests were made with this tube at a constant pressure of 250 pounds, temperatures varying between 500° and 750° C., and rates of feed between 12 and 25 gallons per hour.

A feature of interest is the fact that the percentage recovery of oil is higher for the same temperatures, pressures, and rates of feed than for either the 8-inch by 10-foot or the 8-inch by 14-foot tubes. This indicates clearly that a lesser degree of conversion took place, an assumption which is borne out by the decreased yields of benzene and toluene for the given conditions of operation. The results were mostly of a negative character.

The greatly decreased capacity of the tube as compared with those of greater length made it apparent that the 7-foot tube was too short for commercial purposes, although the reaction was proved to take place readily therein.

GENERAL CONCLUSIONS REGARDING DIMENSIONS OF TUBES.

The results of the experiments for which data have been given show that conversion into low-boiling aromatic hydrocarbons will take place in tubes of any size from 1½ inches by 3 feet to 8 inches by 14 feet.

Reactions occur as readily in one size of tube as another. The variation in extent of heating surface, however, makes it necessary properly to adjust conditions of temperature, pressure, and rate of feed in order to obtain maximum conversion, and minimum waste in the form of ultimate decomposition products. The desirable combination of conditions must be experimentally determined for each oil used as original material. It was found that for an 8-inch diameter 14 feet exceeded the optimum length which could be used, because of the mechanical rather than chemical disadvantages. Likewise it was found that a 7-foot tube was too short, not because of chemical or mechanical difficulties, but on account of the commercial factor of greatly decreased capacity. The most efficient length for tubes 8 inches in diameter was determined to be 10 feet (for the heated zone).

There is no reason for assuming on the basis of the preceding experiments that tubes of larger diameter can not be successfully used. As a matter of fact, tubes 12 inches and 18 inches in diameter were cast and a new design of furnace worked out to accommodate either one of these larger tubes. Because of the necessity of turning out product, experimentation with the larger tubes was discontinued and attention concentrated on the 8-inch tubes, which had proved to develop a fair capacity and to give satisfactory conversion results. Therefore, in the large furnaces which were constructed for operating purposes, 8-inch tubes have been used and data are not available for the larger sizes. The indications are, however, that these larger sizes will be found to work just as efficiently. It seems possible that in the use of these larger sizes, particularly the 18-inch diameter, the addition of an inner core of smaller size would be desirable, as the heat would not have to penetrate to the axis of the tubes and the gases would be confined between the inner and outer tubes so as to gain the full advantage of the greatly increased contact surface. Some idea of what the increase in diameter to 12 and 18 inches means may be gained from the statement that the jump in diameter from 8 to 10 inches will give an increase of 60 per cent in volume, with a resulting gain in feed capacity.

In order to indicate more clearly the gain in capacity resulting from increase in diameter or length, results for the various sizes of tubes are given in Table 55. From this table it will be noted that an increase of 2 inches in diameter and 4 feet in length doubles the capacity. It is to be regretted, therefore, that opportunity has not been afforded for trying out either the 12 or 18 inch tubes.

TABLE 55.—*Relative rates of oil feed in tubes of varying diameters and lengths at a constant temperature and pressure.*

Size of tube.		Rate of feed, gallons per hour.
Diameter.	Length.	
Inches.	*Feet.*	
1.5	3	0.25
4.5	8	8.00
4.5	10	10.00
6.0	9	14.00
6.0	10	17.00
8.0	7	12.00
8.0	10	22.00
8.0	14	30.00

Efficiency has been found to increase with enlargement of the tube diameter and it is reasonable to anticipate, on the basis of the experimental evidence thus far adduced, that further increase in efficiency may result from a still greater increase in diameter.

EXPERIMENTS WITH BATTERIES OF 10 TUBES 8 INCHES IN DIAMETER AND 14 FEET LONG.

As soon as a sufficient number of runs had been made with the single 8-inch tubes to demonstrate the feasibility of carrying out the reaction in a tube of this diameter, a decision was reached on the part of the Aetna company to install six furnaces containing batteries of 10 tubes each. The individual tubes were to be 8 inches in external diameter and 14 feet in over-all length, as the construction of the furnace necessarily reduced the effective length and it was considered that this length would bring the parts of the tubes in which the reaction was substantially accomplished relatively near the favorable limits found by experimentation.

The construction of furnaces on such a large scale before a single furnace was carefully tried out did not seem entirely justified from the experimental point of view, but the desire on the part of the Aetna company for immediate production convinced it of the desirability of the installation. With the construction of these furnaces, attention was naturally concentrated upon their operation. As a result opportunity for further experimentation with types of furnace construction and possible enlargement of the tubes or cracking chambers was limited.

PROBLEMS ENCOUNTERED IN OPERATING FURNACES.

It was soon found that with these large-size furnaces new mechanical and operating problems had been introduced which required solution before reasonable efficiency could be attained. It had been deemed advisable in the design and construction of the furnaces to operate each tube as a separate unit with individual oil feed and condensers, so that the reactions in each tube could be studied separately and conclusions drawn therefrom. The design of the furnace made it impossible to continue the use of pyrometers attached to the tubes themselves, and in order to properly judge the heat conditions in the combustion chamber it became necessary to rely on pyrometric readings of the temperatures existing in the combustion chamber as a whole, and on optical determinations of the temperature that were based on the colors of the brickwork and the tubes themselves.

The intense heats in the furnace chamber made the use of base-metal pyrometers highly unsatisfactory, the pyrometers lasting only a few days before they had to be renewed. The use of asbestos and other insulating material for the protection of pyrometer rods was attempted, but this insulation was soon destroyed and the rods were rendered unfit for further use. In consequence chief reliance was placed on optical readings during the greater portion of time covered by the series of runs for which data are hereinafter given.

After the abandonment of the pyrometer rods as an index of temperatures in the combustion chamber, considerable difficulty was experienced at the outset in obtaining proper heating control. As was to be expected at the beginning of operations, the furnaces could not be continuously operated throughout a period of 24 hours. The frequent shut-downs necessitated the reheating of the furnaces in order to obtain a proper cracking temperature in the tubes, and, more often than not, resulted in overheating. This overheating brought about a great many mechanical difficulties, some of which, for the time being, were of a very troublesome character.

First and more important of these difficulties was the formation of masses of dense, hard coke in the interior of the tubes that completely plugged them and prevented the passage of the hydrocarbon gases. Next in order of importance was the trouble caused by the excessive heating of tubes during the period preparatory to each run in which the furnaces were brought up to the necessary temperature. This trouble manifested itself chiefly in the opening out or bulging of the tubes during the progress of operations.

The mechanical stirring rods were also found to give considerable trouble in the new type of installation. It was found difficult to obtain a rod of sufficient strength to withstand the furnace heats, this trouble also being due to overheating. The rotation of the rods when driven at a considerable rate of speed caused a synchronous rocking of the furnace. The vibration was so great, if the 10 rods were operated simultaneously, that it became necessary to operate only a single stirring rod at a time. This gave opportunity for the building up of coke, or dense, hard carbon on the walls of the tube, as well as on the stirring rod itself, and this deposit was difficult to remove when the rod was again placed in operation.

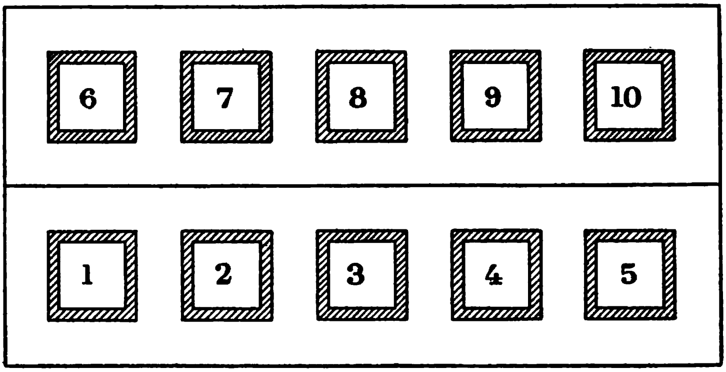


FIGURE 39.—Cross section of tubes in ten-tube furnace.

The most serious difficulty encountered with this type of furnace was, however, the variations in results obtained from different tubes. It was later found that the pumps, which were believed to give an absolutely predetermined rate of feed, did not indicate plugging in the needle valves and instead of a uniform feed in all the tubes, the rate varied from 8 to 30 gallons per hour. As it was thought that the feed mechanism would give no trouble, this handicap was not discovered for some time. It would be unreasonable to expect uniform results with such wide variations existent. The arrangement of the tubes in the furnace and the numbers given to them in operation are shown in figure 39.

Frequently two tubes occupying the same relative position in the furnace, such as, for example, tubes 3 and 8, gave widely varying results, the specific gravities of the recovered oil ranging in some cases from 0.88 to 1.00. The wide differences in character of the results obtained will be more clearly perceived on examination of the data contained in Table 56, which represents the results of a study of the reactions in the individual tubes, extending over a period of some weeks.

TABLE 56.—*Variations in specific gravity of oils recovered from individual tubes in a 10-tube furnace (tubes 8 inches in diameter and 14 feet long).*[a]

Run No.	Specific gravity in tube No.—									
	1	2	3	4	5	6	7	8	9	10
1	0.91	0.98	0.91	0.94	0.89	......	1.00	0.99	0.96	0.92
2	.90	.97	.91	.93	.98	......	......	.95	1.00	.98
3	.92	.94	.96	.96	.92	0.90	.99	.92	.95	.93
4	.92	.96	.99	.92	1.01	.92	.97	.94	.99	.97
5	.94	.94	.90	.94	.94	.94	.98	.95	1.01	.99
6	.93	.98	.99	1.00	.91	.97	1.00	.96	1.01	.96
7	.91	.91	.88	1.00	.89	.96	92	.91	.97	......
8	.94	.89	.96	.87	.93	.95	.87	.91	.98	......
9	.94	.99	.92	.95	1.00	.98	.88	.94	.97	......
10	.93	.87	.89	.98	.97	.92	.90	.97	.96	.92
11	.93	.89	.93	.93	.95	.88	.94	.90	1.06	.98
12	.87	.90	.92	.93	.93	.90	.94	.92	.93	.89
13	.92	.90	.96	1.00	.99	.93	.94	.91	.88	.93
14	.96	.89	.96	99	1.10	.92	.99	.89	.95	92
15	.96	.98	.96	.98	1.02	.96	.93	.90	.93	.94
16	.93	88	.90	1.06	.91	90	.84	.98	1.04	.93
17	.90	.89	.87	.94	96	.90	.98	.92	.98	.97
18	.95	.91	.89	.88	.96	.85	.89	.95	.99	96
19	.92	97	.88	.89	.89	.95	.98	.91	.99	.95
20	.92	.95	.89	.93	.95	.94	.93	.94	1.00	.95
21	.90	.94	.86	.96	.92	.94	.91	1.00	.95	.91
22	1.00	1.01	.95	.97	.91	.98	.97	.99	.99	.95
23	.94	.98	.90	.95	.94	.97	.98	1.01	.99	.94
24	.91	.94	.87	.98	.90	.94	.94	.97	.96	.94
25	.91	.92	.87	.94	.91	.93	.95	.96	.93	.91
26	.98	.99	.97	.99	.97	.96	.97	1.01	1.01	.93
27	1.00	1.02	.92	.95	1.00	.94	.95	1.05	1.05	.96
28	.91	.92	.87	.94	.91	.91	.93	.96	.93	.91
29	.98	.99	.97	.99	.99	.97	.96	1.01	1.01	95
30	1.00	1.02	.92	.95	.95	1.00	.94	1.05	1.06	.98

[a] Compare with results presented in Table 58.

On account of the fact that the principal commercial development of the benzene-toluene process has been in connection with these 10-tube furnaces considerable space has been given in a subsequent part of this report to a description of the mechanical features connected with this type of installation.

RESULTS LOW BECAUSE OF INADEQUATE CONDENSATION.

In considering the percentage yields reported, too much emphasis can not be placed upon the fact that adequate condensation has not been afforded and that on this account the results are less favorable than would be obtained normally.

It is the custom among oil refiners to allow 1½ to 2 square feet of condensing surface per barrel of oil treated in a given retort. On this basis the area of condensing surface for the individual tubes was insufficient and has been detrimental to the products obtained, as is evident from the following facts:

Benzene boils at a temperature of 80° C. and toluene at 110° C. At 30° C. the vapor pressure of benzene is one-fourth atmosphere, at 60° C. it is one-half atmosphere, and at 120° C. it is three atmospheres. The same relative order of temperature and pressure values holds for toluene.

In testing the temperature of recovered oil as it left the condenser figures for an average of six runs ranged between 88° and 125° C. In other words, the liquid left the condenser at a temperature higher than the boiling points of the products sought. This was possible owing to the presence of quantities of higher-boiling constituents which tended to retain a part of the lower-boiling constituents.

Separate condensers were installed, as previously mentioned, in order to collect the liquid products of the gaseous reaction in each individual tube. It has been the practice to tap these condensers and to collect and measure the product in a bucket, the total product from each tube being kept separate from the products of the other tubes. This was done primarily for the purpose of studying the furnace conditions with respect to the individual tubes, and provision was made from the outset for ultimately connecting each primary and secondary condenser allotted to each tube into a common header. It was felt inadvisable to do so as long as the wide variations existed which are shown in Table 56.

The recovered products taken from the condenser at this high temperature evaporated when exposed to the air. Fully 50 per cent of the benzene and toluene were dissipated in this way. This serious loss renders all the reported yields proportionately low. There is absolutely no question in the mind of the authors, or of those in connection with the development operations, but that the yields thus far obtained could have been approximately doubled had adequate condensation been had from the outset.

The inadequacy of the condensing apparatus may be better realized when it is stated that the area of condensing surface per tube was approximately 10 square feet, and the volume of gases per hour passing through the condenser was 1,500 cubic feet. In other words 1 square foot of condensing surface was available for each 150 cubic feet of gas generated per hour.

The foregoing is not intended as a criticism of the operating methods followed, as it was absolutely essential that opportunity be afforded to analyze the products from each tube, which could not have been done in any other manner. It should be borne in mind that the results of experiments extending over a period of seven months are here recorded and that at the outset, and throughout the greater part of the development period, attention was concentrated more on the mechanical problems incident to large-scale operation than on the maximum recovery of conversion products. It was at all times recognized that such a recovery could be obtained, but until the mechanical difficulties had been overcome, and continuous operation attained, the process could not be considered as commercially practicable.

The process could be considered a success, however, when a state of comparatively continuous operation and efficient conversion had been reached, even though efficient condensation, heat economy, preheating of oil, and other standard refinements had not been installed. Common criticism was raised against permitting the gases formed to go into the air instead of burning them, the advisability of which was never questioned. Necessity demanded, however, that more important fundamental considerations be considered first. The matter of the installation of an efficient condenser and scrubber was considered a refinement to be taken up at a later date after the basic mechanical difficulties had been measurably overcome. With the large condenser and numerous other refinements being installed to handle the product from all the furnaces, the yields obtained will show a marked improvement over the ones presented in this bulletin.

The above explanation must be kept constantly in mind in passing on the results recorded, as otherwise entirely erroneous impressions will be formed concerning the availability of the process for general use under normal conditions.

CARBON DEPOSITION.

Throughout the operations for which results are recorded in Table 58 an Oklahoma crude oil was used. The semiasphaltic character of this oil caused a very large percentage of carbon formation, and its use was discontinued in favor of a distillate, from Pennsylvania crude, with a boiling range between 250° and 400° C. This does not imply that Oklahoma oils are unsuitable for use in the process, but means that it is desirable first to remove the gasoline fraction and the heavy residuum. Laboratory tests have shown clearly that if this were done an Oklahoma oil would probably give more satisfactory results than the Pennsylvania distillate which has since been used, because the asphaltic properties of some of its constituents favor splitting into benzene and its homologues.

The lack of distillation facilities at the plant, together with the cost thereof, as compared with the price at which a distillate could be purchased at local refineries, caused the abandonment of the Oklahoma crude oil in favor of this distillate.

The total amount of carbon formed, on the basis of oil used, has been rather roughly determined by scraping the carbon from the carbon receptacles into wheelbarrows and weighing it. It was found to average between 1 and 1¼ pounds per gallon of oil used. This means that for every 1,000 gallons of oil the process made and cared for between 1,000 and 1,500 pounds of carbon. The ability to successfully handle this large amount of carbon is believed to constitute a unique difference between this and other cracking processes. The total amount of carbon formed while using Oklahoma crude oil is greatly in excess of the amount formed from the distillate subsequently used. The latter oil averaged only one-fourth to one-half pound per gallon. This indicates that a considerable quantity of the total carbon formed came from the heavy bottoms, or residuum, absent in the distillate.

REDUCTION IN LENGTH OF TUBES.

It was felt that the mechanical troubles could largely be remedied by shortening the tubes. Accordingly, in later experiments it was decided to replace the 14-foot tube by tubes 11½ feet long, giving an effective heating length of approximately 10 feet. Several weeks were consumed in the reconstruction of the furnaces to accommodate the shorter tubes and in the preparation of drawings and the casting of new furnace tops to replace those formerly used. The experiments with 8-inch tubes 14 feet long in batteries of ten brought the same conclusion which had been reached regarding their use singly—that for purely mechanical reasons a length of 14 feet was too great.

RESULTS OBTAINED.

The results of operations in the large 14-foot furnaces are given in Table 57, and represent the results of more than three months of experimentation and operation. Space would not permit the inclusion of all the data for the six furnaces; consequently selection has been made of typical runs and results.

It has been impossible to indicate the temperatures existing for the reason that these varied all the way from 600° C. up to 800° C., and precise determinations were not obtainable by optical readings. The temperatures that were reported represented only the best guesses of the heaters (furnace tenders) or the engineers in charge of operations, and therefore no good purpose would be served by including them in the table.

TABLE 57.—*Benzene and toluene formed from cracking Oklahoma crude oil in 10-tube furnaces (tubes 8 inches in diameter and 14 feet long) at varying pressures and rates of oil feed.*[a]

[Temperatures varied from 600° C. to 800° C., and were determined by judging the color of the tubes, aided in some cases by pyrometer inserted in checker work of furnace. In determining specific gravities for runs in multiple-tube furnaces, covering quantities ranging from 1 to 1,500 gallons, the values indicated are correct within 0.02.]

Pressure per square inch.	Rate of oil feed per hour.	Oil used.	Oil recovered.	Specific gravity of oil recovered.	Benzene in recovered oil.	Toluene in recovered oil.	Total benzene and toluene in recovered oil.	Benzene formed on basis of original oil.	Toluene formed on basis of original oil.	Total benzene and toluene formed on basis of original oil.
Pounds.	*Gallons.*	*Gallons.*	*Per cent.*		*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
100	18	1,334	38	0.92	7.9	5.3	13.2	3.0	2.0	5.0
100	18	1,343	34	.94	8.5	7.9	16.4	2.9	2.7	5.6
150	23	1,461	37	.93	8.7	7.0	15.7	3.2	2.6	5.8
150	25	1,829	25	.96	12.8	8.8	21.6	3.2	2.2	5.4
150	27	866	27	.97	12.6	9.6	22.2	3.4	2.6	6.0
175	26	1,114	31	.94	11.6	10.0	21.6	3.6	3.1	6.7
175	26	1,286	32	.94	11.5	10.6	22.1	3.8	3.5	7.3
175	26	1,360	37	.93	10.8	8.9	19.7	4.0	3.3	7.3
175	26	1,365	37	.91	8.7	6.2	14.9	3.2	2.3	5.5
175	26	1,393	31	.93	10.3	9.4	19.7	3.2	2.9	6.1
175	27	961	26	.94	11.9	9.2	21.1	3.1	2.4	5.5
200	27	1,363	30	.91	9.7	7.7	17.4	2.9	2.3	5.2
200	28	1,494	25	.93	12.8	9.6	22.4	3.2	2.4	5.6
250	30	200	33	....	15.1	7.6	22.7	5.0	2.5	7.5
250	30	200	33	....	15.1	7.6	22.7	5.0	2.5	7.5
250	30	210	21	....	30.9	10.5	41.4	6.5	2.2	8.7
250	30	240	33	....	18.8	7.0	25.8	6.2	2.3	8.5
250	30	252	28	....	19.6	6.8	26.4	5.5	1.9	7.4
250	30	333	31	.93	13.9	10.0	23.9	4.3	3.1	7.4
250	30	333	25	....	24.0	8.8	32.8	6.0	2.2	8.2
250	30	363	23	....	28.0	10.9	38.9	6.2	2.5	8.7
250	30	382	24	....	27.1	9.0	36.1	6.5	2.3	8.8
250	30	387	26	....	23.8	7.7	31.5	6.2	2.0	8.2
250	30	453	20	.96	17.5	8.0	25.5	3.5	1.6	5.1
250	30	460	25	....	26.0	10.4	36.4	6.5	2.6	9.1
250	30	483	33	....	18.8	6.7	25.5	6.2	2.2	8.4
250	30	512	32	....	20.3	7.5	27.8	6.5	2.4	8.9
250	30	536	22	....	25.4	10.4	35.8	5.6	2.3	7.9
250	30	580	30	....	20.0	7.7	27.7	6.0	2.3	8.3
250	30	634	19	....	34.7	11.6	46.3	6.6	2.2	8.8
250	30	667	24	....	23.3	7.9	31.2	5.6	1.9	7.5
250	30	725	26	....	23.1	7.7	30.8	6.0	2.0	8.0
250	30	762	26	....	24.2	8.9	33.1	6.3	2.3	8.6
250	30	884	24	.94	12.9	10.0	22.9	3.1	2.4	5.5
250	30	1,070	27	....	25.2	8.9	34.1	6.8	2.4	9.2
250	30	1,072	22	....	27.3	9.6	36.9	6.0	2.1	8.1
250	30	1,137	22	.96	10.0	10.0	20.0	4.4	2.2	6.6
250	30	1,490	24	....	25.0	8.8	33.8	6.0	2.1	8.1
250	30	1,529	26	....	24.2	8.8	33.0	6.3	2.3	8.6

[a] Results low, due to insufficient condensation.

It will be noted that with the Oklahoma crude oil, the percentage of benzene formed exceeds on an average the percentage of toluene formed. This is in sharp contrast with later results, obtained with the use of the distillate oil, in which the percentage of toluene exceeds that of benzene. (See page 165.)

The yields of benzene and toluene shown in this table are, on the whole, less than the yields obtained with the same oil in the single-tube furnaces. The wide variation both in the specific gravities and in the total amounts of benzene and toluene formed is to be explained by the different temperatures which existed during the course of the runs. Heat control is of course the most vital factor in the process and the inability to obtain uniform temperature conditions in the series of runs reported is responsible for the variations to which attention has been called.

EXPERIMENTS WITH BATTERIES OF TEN TUBES 8 INCHES IN DIAMETER AND 11½ FEET LONG.

As has been pointed out, the decision to reduce the length of the tubes in the 10-tube furnaces from 14 feet to 11½ feet was due to a desire to reduce mechanical difficulties incident to the operation of the longer stirring rods. It was decided at the same time to decrease the rate of oil feed, in order to permit using a lower temperature for the same relative amount of conversion.

It was determined also to conduct experiments for the purpose of ascertaining whether the pressure previously used (250 pounds per square inch) could not be materially reduced in view of the slower rate of feed. An extensive series of experiments demonstrated that with the distillate a pressure of 150 to 175 pounds at the reduced feed gave more satisfactory results than the higher pressures.

The reduction in the length of the tubes from 14 feet to 11½ feet gave an effective furnace length of approximately 10 feet, which is the optimum length as determined by a series of experiments with the single-tube furnaces for an 8-inch diameter. The results with the shorter tubes have shown an improvement over those obtained with the longer tubes. The average recovery of aromatic hydrocarbons has been slightly larger, but the greatest improvement has been with respect to the mechanical difficulties that were previously encountered. The shortening of the stirring rod has lessened the strain to which the rod was subjected and has prolonged its life in service.

The same variation among different tubes in the results of the conversion in each individual tube has persisted, however, as is shown by the specific gravities of the recovered oil, given in Table 58. The specific gravities for a single run varied all the way from 0.82 to 1.01. The average of the specific gravities of the oils from the ten tubes may be 0.93, yet the specific gravity of the oil from one or more tubes may be as low as 0.82 and from other tubes as high as 1.05. This means that there will be a larger percentage of impurities in the form of olefins and paraffins in the recovered product, which can not be removed by simple distillation. Information is now available to the effect that in the nitration of the products impurities can be readily removed, thus making their presence of little moment. In fact, recovered oils of even lower specific gravities would be just as satisfactory for nitration, which would make the control conditions still easier of attainment. Table 58 follows:

TABLE 58.—*Variations in specific gravity of oils recovered from individual tubes in a 10-tube furnace (tubes 8 inches in diameter and 11½ feet long).*[a]

Run. No.	Specific gravity of oil recovered from tube No.—									
	1	2	3	4	5	6	7	8	9	10
1	1.00	0.94	1.01	0.96	....	0.95	0.84	....	0.97	0.96
2	.94	.96	.96	.94	....	.89	.94	....	.96	.91
3	.84	.93	.88	.92	1.02	.98	.98	0.90	.99	1.01
4	.92	.97	.93	1.01	.86	.84	.87	.87	.95	.88
5	.94	.94	.91	.92	....	.95	.93	....	.90	.86
6	....	.98	.96	.99	....	1.00	.99	.96	1.09	....
7	.84	.93	.88	.92	1.02	.98	.98	.90	.99	1.01
8	.92	.97	.93	1.01	.86	.84	.87	....	.95	.88
9	.94	.94	.91	.92	....	.95	.93	....	.90	.86
10	.92	.86	.85	.89	....	.86	.85	....	.90	.97
11	.97	.98	.96	.95	1.02	.98	.94	.91	.96	....
12	.97	.97	.94	.94	.92	.94	.98	1.00	.96	.96
13	.97	.97	.98	.95	.95	.95	.91	1.05	....	.95
14	.93	1.03	.97	.99	.97	1.03	.95	1.01	....	.99
15	....	.99	.99	.96	.95	.91	.94	1.00	1.07	1.02
16	.99	.96	.97	1.00	.98	1.00	1.04	1.00	1.06	1.01
17	1.06	.97	.97	.98	1.04	.97	.97	1.07	1.02	1.00
18	.96	1.05	1.03	1.00	.98	1.06	.97	1.06	1.03	....
19	.98	.92	.91	.96	.93	1.00	.90	.91	.95	.97
20	.90	.96	.96	.96	.97	.97	1.03	.97	.98	.91
21	.90	.96	.91	.96	.95	.97	.92	.97	1.03	.94
22	.88	.91	.92	.87	.88	.89	.92	.91	.92	.90
23	.92	.95	.95	.91	.94	.95	.96	.97	.98	.97
24	.89	.90	.89	.89	.88	.94	.93	.95	.98	.95
25	.96	.92	.94	.90	.88	.98	.92	1.00	....	.92
26	.95	.93	.93	.95	.90	1.00	.95	1.00	.97	.91
27	.90	.92	.92	.89	.91	.95	.97	.99	....	.96
28	.86	.89	.92	.90	.89	.88	.93	1.02	.96	.98
29	.90	.95	1.00	1.05	.98	.91	1.00	1.03	1.03	1.04
30	.87	.82	.97	.97	.93	.97	.92	.99	1.01	.93

[a] Compare with data given in Table 56.

The significance of the varying specific gravities of products yielded by the individual tubes is more clearly indicated by an examination of Tables 59, 60, and 61, which show the percentages of benzene, toluene, and total benzene and toluene formed, on the basis of original oil, in the same runs for which specific gravities are given in Table 58 for the 8-inch by 11½-foot furnaces. The average recovery of benzene varied from 0.5 to 3 per cent, whereas the percentage of toluene varied from 1 to 4 per cent. The substitution of a distillate oil for the Oklahoma crude oil that was previously used has made no difference in the variation in results, and it is evident that the difference in the nature of the oils has no bearing on the problem. The problem is purely one of heat control. Experience has shown that proper heat control is relatively easy in a single-tube furnace, but more difficult in the multiple-tube furnaces. The tables showing percentages of products obtained follow:

TABLE 59.—*Variations in percentage of benzene formed in individual tubes in a 10-tube furnace (tubes 8 inches in diameter and 11½ feet long), calculated on basis of original oil.*[a]

Run No.	Percentage of benzene formed in tube No.—									
	1	2	3	4	5	6	7	8	9	10
1	3.5	3.0	3.6	2.8	......	5.8	0.8	......	4.6	3.1
2	3.2	4.2	3.3	2.3	......	2.1	2.9	......	3.5	1.8
3	1.0	3.1	2.1	2.5	3.1	3.1	3.8	2.7	2.8	0.7
4	2.0	2.7	2.8	1.2	1.7	2.3	1.4	......	1.6	0.9
5	3.2	2.6	2.8	1.8	2.6	2.3	2.9	......	2.4	3.4
6	......	3.4	2.2	1.3	......	2.8	2.9	2.9	1.3	......
7	1.0	3.1	2.1	3.1	3.1	3.8	2.7	2.8	0.7	2.5
8	2.0	2.7	2.8	2.2	2.7	2.3	1.4	......	1.6	0.9
9	3.2	3.6	2.8	2.8	......	2.3	2.9	......	3.4	3.4
10	3.8	1.8	1.6	2.4	......	1.8	1.0	......	1.2	1.5
11	4.2	4.0	5.3	5.8	2.0	4.2	3.5	3.6	2.4	3.6
12	......	3.9	3.9	2.8	3.8	2.5	2.0	3.1	3.8	4.0
13	2.4	3.8	2.4	2.7	3.1	3.3	2.1	2.5	......	4.4
14	2.0	3.6	3.8	4.3	3.8	3.5	3.8	2.9	......	2.0
15	2.4	4.2	3.9	2.9	2.4	3.0	3.5	2.8	......	3.0
16	3.1	2.9	2.8	3.6	3.9	2.0	3.9	3.0	1.4	2.3
17	3.6	3.4	2.6	3.1	3.0	3.2	3.5	2.0	1.9	3.2
18	2.7	3.8	3.6	3.0	2.5	2.9	3.3	4.5	2.5	2.0
19	3.2	3.6	2.7	3.0	3.0	3.8	2.8	3.0	2.6	3.1
20	2.9	3.0	2.4	2.5	1.8	2.4	3.2	1.9	2.2	2.6
21	2.5	3.4	3.3	3.2	3.1	4.7	3.4	4.2	4.5	2.4
22	2.4	3.0	3.0	2.4	2.3	1.8	3.7	2.8	2.4	2.5
23	3.1	2.9	3.1	3.0	2.9	3.3	3.3	3.1	2.8	2.7
24	2.9	2.7	3.3	2.6	3.5	4.2	3.6	2.4	2.9	2.4
25	3.1	3.2	3.7	3.3	3.5	2.8	3.2	2.9	......	3.5
26	3.0	2.9	2.9	4.6	2.4	2.8	3.5	2.9	......	3.0
27	3.0	2.6	2.7	3.8	3.3	3.1	2.9	2.2	......	3.0
28	2.6	3.0	3.2	2.7	2.4	2.3	4.0	2.9	3.4	2.6
29	2.6	3.3	3.4	2.9	2.9	2.1	2.3	2.3	1.5	2.1
30	2.5	2.6	2.6	2.3	2.6	2.4	2.4	3.0	2.1	1.2

[a] Results low, due to insufficient condensation.

TABLE 60.—*Variations in percentage of toluene formed in individual tubes in a 10-tube furnace (tubes 8 inches in diameter and 11½ feet long), calculated on basis of original oil.*[a]

Run No.	Percentage of toluene formed in tube No.—									
	1	2	3	4	5	6	7	8	9	10
1	3.5	3.1	3.6	2.9	......	5.4	0.9	......	4.5	3.4
2	3.8	4.3	3.5	2.5	......	2.2	3.6	......	4.0	1.7
3	1.3	3.6	2.7	3.1	3.2	3.7	4.2	3.2	3.2	2.8
4	2.3	3.0	3.0	1.3	2.0	3.8	1.6	......	1.4	1.9
5	3.2	2.6	2.8	1.8	2.3	2.9	2.4	......	2.4	3.4
6	......	3.4	2.4	1.6	......	2.8	3.0	3.1	1.3	......
7	1.9	3.6	2.7	3.1	3.2	3.3	4.2	3.2	2.9	0.9
8	2.3	3.0	3.0	1.3	2.0	3.8	1.6	......	1.4	0.9
9	3.6	3.0	2.8	2.4	......	2.4	3.0	......	2.3	3.2
10	3.5	2.3	1.2	2.3	......	1.0	0.9	......	1.0	1.4
11	4.4	4.2	5.7	3.3	1.1	4.2	2.6	3.8	3.8	2.5
12	......	3.9	3.9	2.8	3.8	3.8	2.4	2.0	2.1	3.8
13	2.5	4.2	3.8	3.2	3.6	2.6	2.6	2.6	......	4.4
14	2.4	3.8	4.2	5.1	4.0	2.8	3.4	3.1	......	2.2
15	2.6	4.6	3.7	3.3	2.7	2.5	3.7	2.9	......	3.6
16	3.2	3.1	2.9	2.7	3.2	2.1	3.1	2.2	1.5	2.5
17	2.8	3.7	3.0	3.5	3.1	2.3	3.8	4.2	2.2	2.4
18	2.8	4.0	2.8	3.1	2.6	2.9	3.6	4.5	2.5	2.6
19	3.4	4.2	3.2	3.4	3.6	3.9	3.7	3.5	3.0	3.8
20	3.2	3.2	2.6	2.7	2.9	2.5	2.4	3.3	1.4	2.8
21	3.1	3.5	2.3	2.5	3.8	4.9	2.8	2.5	2.4	2.7
22	3.5	4.1	3.8	4.3	3.8	2.8	4.7	4.0	2.8	3.8
23	4.4	3.7	3.8	4.4	4.1	2.6	3.9	2.7	3.3	3.1
24	4.2	3.7	3.9	2.0	4.8	4.7	3.8	3.1	3.2	3.9
25	3.8	3.9	4.0	4.0	4.6	2.9	3.5	3.6	......	3.8
26	3.3	3.3	3.6	5.5	3.1	2.8	3.9	3.1	......	3.2
27	3.0	2.6	2.7	3.8	3.3	3.1	3.9	2.2	......	3.1
28	3.9	3.7	4.0	3.4	3.5	3.4	4.2	3.1	3.6	3.0
29	3.0	3.4	3.5	3.0	2.4	2.3	2.5	2.5	2.2	0.9
30	3.4	2.9	2.8	2.4	3.0	2.6	2.8	3.1	1.3	1.4

[a] Results low, due to insufficient condensation.

TABLE 61.—*Variations in total percentage of benzene and toluene formed in individual tubes in a 10-tube furnace (tubes 8 inches in diameter and 11½ feet long), calculated on basis of original oil.*[a]

Run No.	Percentage of benzene and toluene formed in tube No.—									
	1	2	3	4	5	6	7	8	9	10
1	7.0	6.1	7.2	5.7	......	11.2	1.7	......	9.1	6.5
2	7.0	8.5	6.8	4.8	......	4.3	6.5	......	7.5	3.5
3	2.3	6.7	4.8	5.6	6.3	6.8	8.0	5.9	6.0	2.6
4	4.3	5.7	5.8	2.5	3.7	6.1	3.0	......	3.0	2.8
5	6.4	5.2	5.6	3.6	4.9	5.2	5.3	......	4.8	6.8
6	......	6.8	4.6	2.9	......	5.6	5.9	6.0	2.6	......
7	2.9	6.7	4.8	6.2	6.3	7.1	5.9	5.7	3.6	3.4
8	4.3	5.0	5.1	4.6	6.3	6.1	3.0	......	2.1	1.8
9	6.8	6.8	5.6	5.1	4.7	4.7	5.9	......	3.0	6.6
10	7.3	5.3	3.9	5.7	......	2.8	1.9	......	5.7	2.9
11	8.6	8.4	11.0	9.1	......	8.4	6.1	7.4	2.2	6.1
12	......	7.8	7.8	5.6	3.1	6.3	4.4	5.1	6.2	7.8
13	4.9	8.0	6.2	5.9	7.6	6.9	4.7	5.1	5.9	8.8
14	4.4	7.4	8.0	9.4	6.7	7.5	6.9	6.0	......	4.2
15	5.0	8.8	7.6	6.2	7.8	5.5	6.4	5.7	......	6.6
16	6.3	6.0	5.5	6.3	5.1	4.1	6.1	5.2	2.9	4.8
17	6.4	7.1	5.6	6.6	7.1	5.5	7.7	6.2	4.1	5.6
18	5.5	6.6	6.4	6.1	6.1	5.8	7.8	9.0	5.0	4.6
19	6.6	7.0	5.9	6.4	5.1	7.7	6.3	6.5	5.6	6.9
20	6.1	6.2	5.0	5.2	6.6	4.9	6.5	5.2	3.6	5.4
21	5.6	6.5	6.1	5.7	4.7	9.6	6.2	6.7	6.9	5.1
22	5.9	6.5	6.8	6.7	6.9	4.6	8.4	6.8	5.2	6.4
23	7.5	7.3	6.9	7.4	6.1	5.9	7.2	6.8	6.1	5.8
24	7.1	6.9	7.5	4.6	7.0	8.9	7.4	5.5	6.1	6.3
25	6.9	7.0	7.7	7.3	8.3	5.7	6.7	6.5	......	7.3
26	6.3	6.2	6.5	10.1	8.1	5.6	7.4	6.0	......	6.2
27	6.0	5.6	5.4	7.6	5.5	6.2	6.8	4.4	......	6.1
28	6.5	6.9	7.7	6.1	6.6	5.7	8.2	6.0	7.0	5.6
29	5.6	6.3	6.9	5.9	5.9	4.4	4.8	4.8	3.7	3.0
30	5.9	6.0	5.4	4.7	5.6	5.0	5.2	6.1	2.5	2.6

[a] Results low, due to insufficient condensation.

The runs shown in Table 61 were all made on the basis of 150 pounds pressure, but at varying rates of feed. No attempt has been made to present all the data for the six furnaces, as they would be merely cumulative in character.

In considering the total recoveries of benzene and toluene, it should be borne in mind that the percentages shown are not the maximum recoveries that can be obtained from a given quantity of oil. These percentages, as has been pointed out, are based on the calculated amount of aromatic hydrocarbons in the distillation cut up to a temperature of 175° C. The residue from the 175° C. cut is highly suitable for rerunning through the furnace, and will yield an amount of aromatic hydrocarbons equal to that recovered by the first run. The result is that from a given quantity of oil the maximum recovery on running the oil twice through the tubes will be at least double that reported here. That part of the cut up to 175° C., above the toluene fraction, is even more suitable for rerunning than the cut above 175°, as the former contains the xylenes and solvent napthas which have been proven to be a most prolific source of both benzene and toluene. In addition, it must be borne in mind that recovery has not been made of the aromatic hydrocarbons mechanically carried in the uncondensed gases. The gases, which in order to maintain uniform pressure were constantly being released during the operation of the process, did not pass through an efficient condenser or scrubber, but into a gas header and thence into a small gas trap, where a portion of their liquid content was removed. These gases mechanically carry over a considerable percentage of the heavier as well as of the lighter oils. Although the gas traps were of great value in indicating qualitatively the amount of liquid product which is mechanically carried by the gases, they did not remove an appreciable quantity of these constituents.

TABLE 62.—*Benzene and toluene formed from cracking a distillate oil in 10-tube furnaces (tubes 8 inches in diameter and 11½ feet long) at a constant pressure of 150 pounds per square inch.*[a]

[For distillation analysis of oil used see Table 50.]

Oil used.	Oil recovered.	Specific gravity of recovered oil.	Benzene in recovered oil.	Toluene in recovered oil.	Total benzene and toluene in recovered oil.	Benzene formed on basis of original oil.	Toluene formed on basis of original oil.	Total benzene and toluene formed on basis of original oil.
Gallons.	*Per ct.*		*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
242	29.2	0.98	11.7	11.7	23.4	3.4	3.4	6.8
274	30.6	.95	11.3	11.6	22.9	3.5	3.6	7.1
329	26.1	.95	12.7	13.0	25.7	3.3	3.4	6.7
368	26.9	.97	13.7	14.5	28.2	3.7	3.9	7.6
481	33.8	.91	9.7	11.2	20.9	3.3	3.8	7.1
576	33.1	.96	12.4	12.9	25.3	4.1	4.3	8.4
578	26.3	.96	12.3	14.6	26.9	3.2	3.8	7.0
590	28.0	.95	12.1	13.9	26.0	3.4	3.9	7.3
990	37.0	.91	8.7	11.1	19.8	3.2	4.1	7.3
1,130	28.0	.94	12.5	13.6	26.1	3.5	3.8	7.3
1,156	32.0	.95	9.4	9.7	19.1	3.0	3.1	6.1
1,170	25.0	.97	14.0	13.6	27.6	3.5	3.4	6.9
1,283	25.6	.97	13.5	14.3	27.8	3.5	3.7	7.2
1,382	37.4	.91	7.6	8.4	16.0	2.8	3.1	5.9
1,561	29.9	.94	9.7	11.0	20.7	2.9	3.3	6.2
1,600	24.1	.98	12.9	13.8	26.7	3.1	3.3	6.4
1,627	30.0	.94	8.0	9.7	17.7	2.4	2.9	5.3
1,855	31.8	.94	11.0	11.3	22.3	3.5	3.6	7.1
1,878	25.2	.96	14.4	14.8	29.2	3.6	3.7	7.3
1,878	25.6	.95	11.9	13.9	25.8	3.1	3.6	6.7
1,884	27.4	.93	10.7	11.8	22.5	2.9	3.2	6.1
2,200	34.0	.92	8.8	10.6	19.4	3.0	3.6	6.6
2,414	29.8	.96	12.0	12.6	24.6	3.6	3.8	7.4
2,439	25.6	.96	11.6	12.3	23.9	3.0	3.2	6.2
2,753	34.5	.92	9.7	11.2	20.9	3.3	3.8	7.1

[a] Results low, due to insufficient condensation.

GENERAL CONCLUSIONS REGARDING MULTIPLE-TUBE FURNACES.

Observations of the results obtained in a single-tube furnace as compared with those obtained in the 10-tube furnaces have demonstrated the superiority of the single-tube over the multiple-tube arrangement.

No difficulty was experienced in maintaining temperature control over the single tubes, whereas it has been a constant problem to obtain anything like uniformity of heating conditions with the multiple-tube furnace. To get a uniform heat over a small area of the tube it must be heated on more than one side by burners near one plane.

This is not possible in the multiple-tube furnace, but is easy of accomplishment in the single-tube type. One side of the tube in the multiple furnace often becomes too hot and the other side too cold. This would not be the case with the smaller installation.

Separate compartments for each tube would, it is believed, be as satisfactory as single-tube furnaces if sufficient combustion space were allowed. The cost of construction would naturally be less if a number of these compartments were constructed as a unit. It is not recommended, however, that more than four such compartments be arranged in a single unit.

Another recommendation in favor of the single tube is the fact that accurate observation of heating conditions is permitted, which is impossible in the multiple type. With the single-tube furnaces a proportionally much larger combustion space can readily be obtained. The larger combustion space will permit a better mixture of the gas and air and will tend to give more uniform heat conditions, owing to the greater distance the products of combustion must travel before reaching the tubes. An outer combustion chamber in which combustion takes place before the gases enter the furnace chamber in which the single tube is set would enable the brickwork in the interior of the multiple furnaces to be dispensed with without any change in the results.

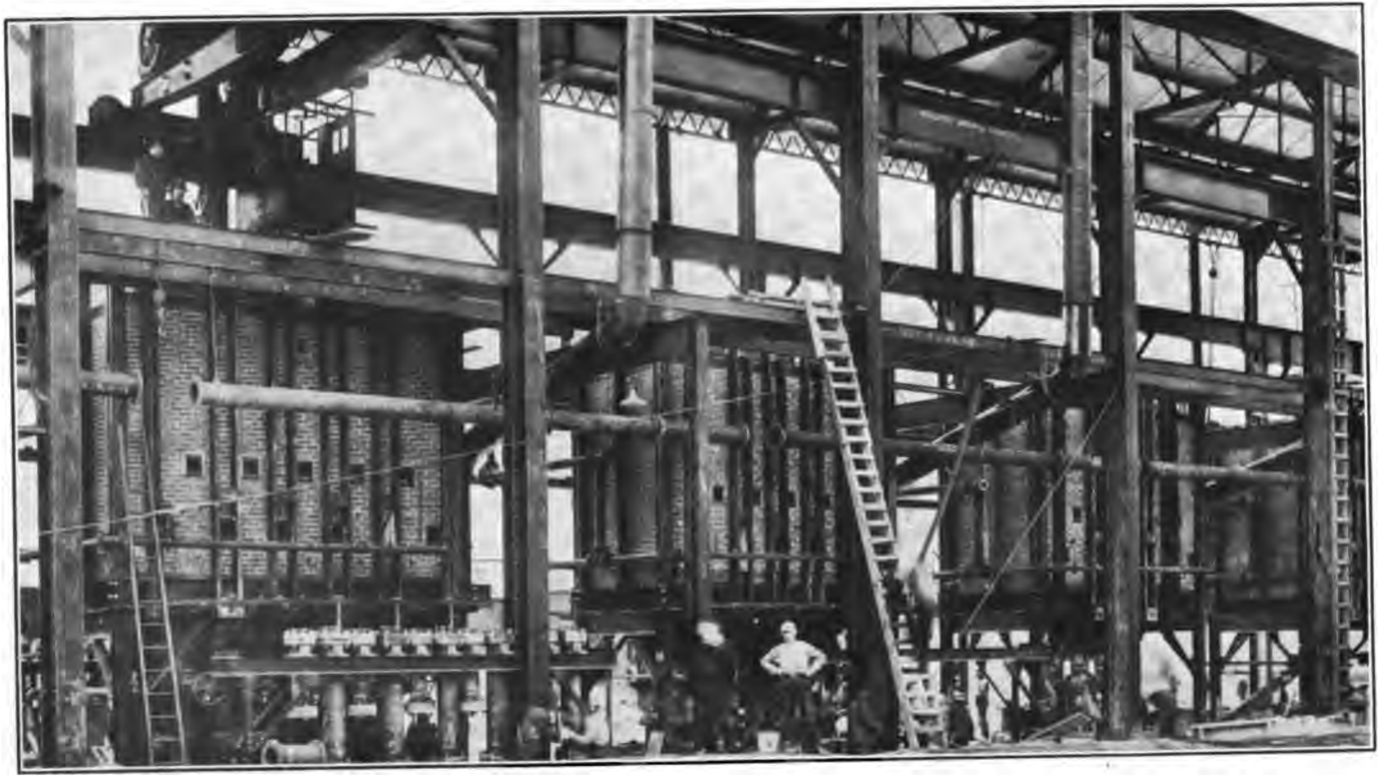
Independent operation of the tubes would permit a tube to be removed or replaced without interfering with the operation of the other tubes. This is difficult in the case of a nest of tubes in a multiple furnace.

DESCRIPTION OF AETNA PLANT AS FINALLY CONSTRUCTED.

FURNACE CONSTRUCTION.

The character of the furnace construction is indicated by Plate III, which shows the furnaces partly completed. The situation of the burners in the furnaces indicated by the portholes. The arrangement of the furnaces in the building and their appearance when completed are shown in Plate I, *B*. The carbon pots attached to the furnace in the foreground were merely a temporary installation. The details of the furnace construction are given in figures 40 and 41, together with the general dimensions of the furnace. The relation of the furnaces to the building construction is indicated by figure 42.

As will be observed from both the plates and the figures mentioned, the furnace proper consists of an outer shell of common brick, lined with a single thickness of standard fire brick. The over-all dimensions of the 10-tube furnace are approximately as follows: Height 12 feet 5 inches, clearance between top and bottom plates 11 feet; length of sides 16 feet 1½ inches, clearance between



CHARACTER OF FURNACE CONSTRUCTION.

inner walls 13 feet 10½ inches; and width 9 feet 4½ inches, clearance between side walls 7 feet 1½ inches. Twenty-two gas burners are used for heating each furnace. Any common type of gas burner of

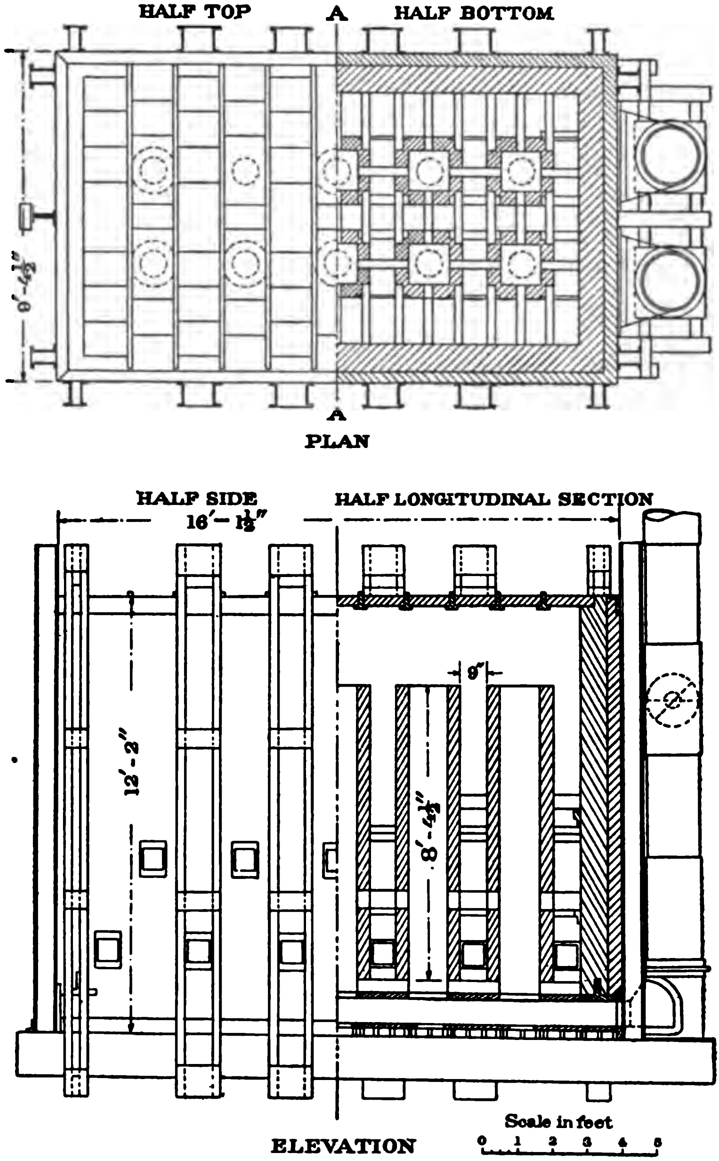
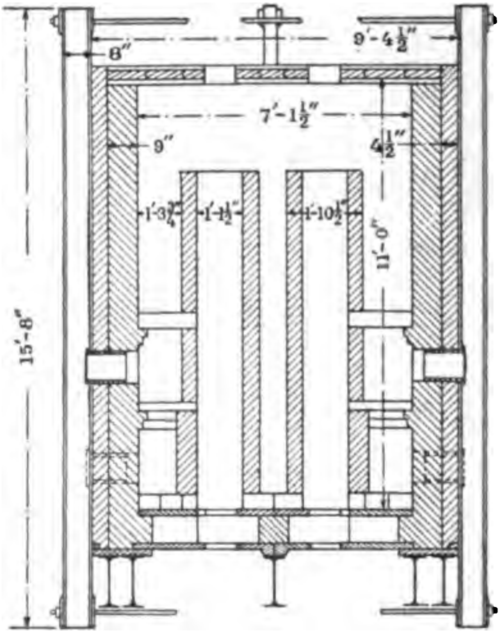


FIGURE 40.—Plan and elevation of furnace at development plant.

sufficient capacity may be used. The consumption of gas per tube averages about 300 cubic feet per hour with the present installation, which has not been operated with particular regard to economic heating.

The interior of the furnace is filled with a checkerwork of fire brick. The gaseous products of combustion pass through a baffle arrangement from the gas ports, ascend through the checkerwork and are drawn inside the chambers of fire brick in which the tubes are placed. From these chambers the gases pass out through two flues at the bottom of the furnace shell and are discharged into the open air through stacks 18 inches in diameter placed at one end of the furnace. The flues are so arranged that the products of combustion of the 11 burners on one side of the furnace pass out through an individual stack. The brick shell of the furnace is strengthened by five steel buckstays on each side and three on each end, these stays being approximately 15 feet 8 inches high and 8 inches wide. The tops of the stays are connected with tie-rods so as to give greater stability to the furnace. The burners of the upper row are placed at a height of 3½ feet above the heater platform, shown in Plate I, *B*. It has been found necessary to have a double set of burners in order to supply the additional heat required to maintain the tubes at the desired temperature.



CROSS SECTION A-A

FIGURE 41.—Cross section of furnace. (See fig. 40.)

The furnaces are set at a height of 10½ feet above the ground, and rest on 16-inch I beams, supported by 8-inch I beams. The arrangement of the supports is fully shown in Plate I, *B*. The furnaces were thus elevated because it was necessary to place carbon receptacles immediately under the bottom of the tubes, and to rotate the stirring rod from below. The carbon receptacles and the stirring-rod drive rest on concrete foundations.

OIL-FEEDING EQUIPMENT.

The general arrangement of the oil feed to the tubes is shown by figure 43, which is a sketch of a furnace and its connections. The oil is pumped to the supply tanks, which feed the five tubes on each side of the furnace and is forced into the tubes by means of triplex pumps, one pump being supplied for each tube.

The connection of the feed lines with the top of the tubes is shown in Plate IV. The oil delivered to each tube by a separate pump traverses a pipe line which is suspended below the upper floor, and thence passes through a needle valve into the top of the tube. The pumps are so constructed that each pump can be calibrated to give any desired rate of flow. The needle valve, which is shown in the foreground, permits independent control of the rate at which the oil is fed into the tube, and enables the oil supply to be immediately cut off in case of accident happening to the tube. The general arrangement of the pumps for the individual furnaces is shown by Plate V, which gives a general view of the upper floor of the furnace building.

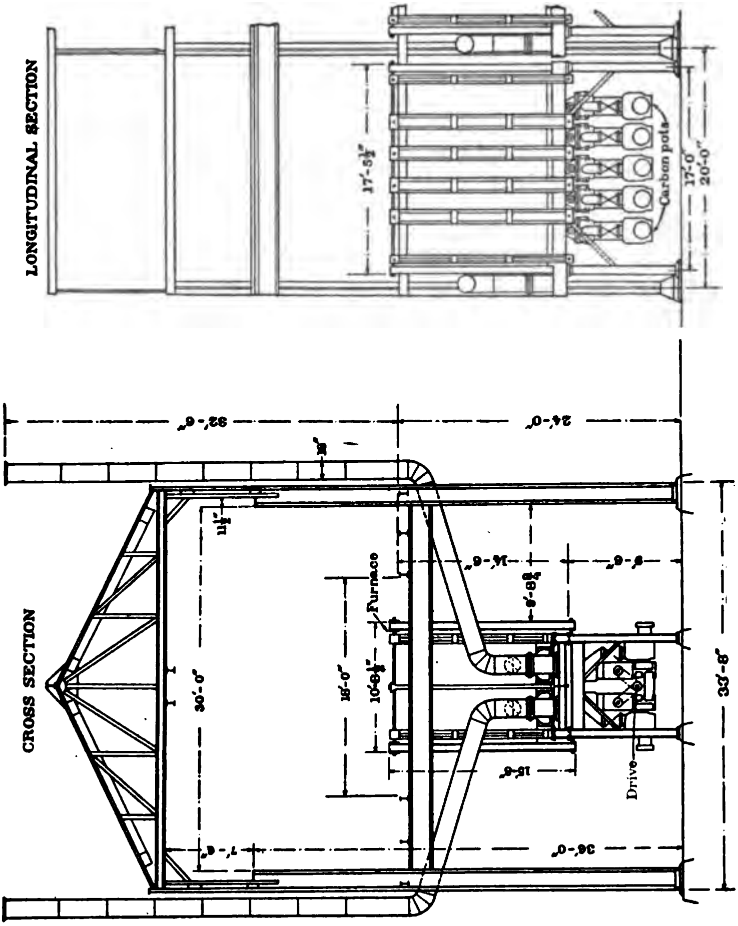


FIGURE 42.—Arrangement of stacks of 10-tube furnaces. Relation of furnaces to building construction.

The short tubes shown in the foreground are metal baskets which are suspended in the top of the cracking tube and serve to spread the oil as it is fed in, thus facilitating immediate vaporization. The basket consists merely of a short tube with a perforated cap or end, cast with the tube. The large hole in the end of this tube serves as a bearing for the stirring rod.

A system of individual pumps for each tube, which in the case of the installation shown comprises 60 pumps for the 60 tubes, is expensive. There is no reason why a single pump supplying oil to a common header should not prove equally satisfactory. The use of such a

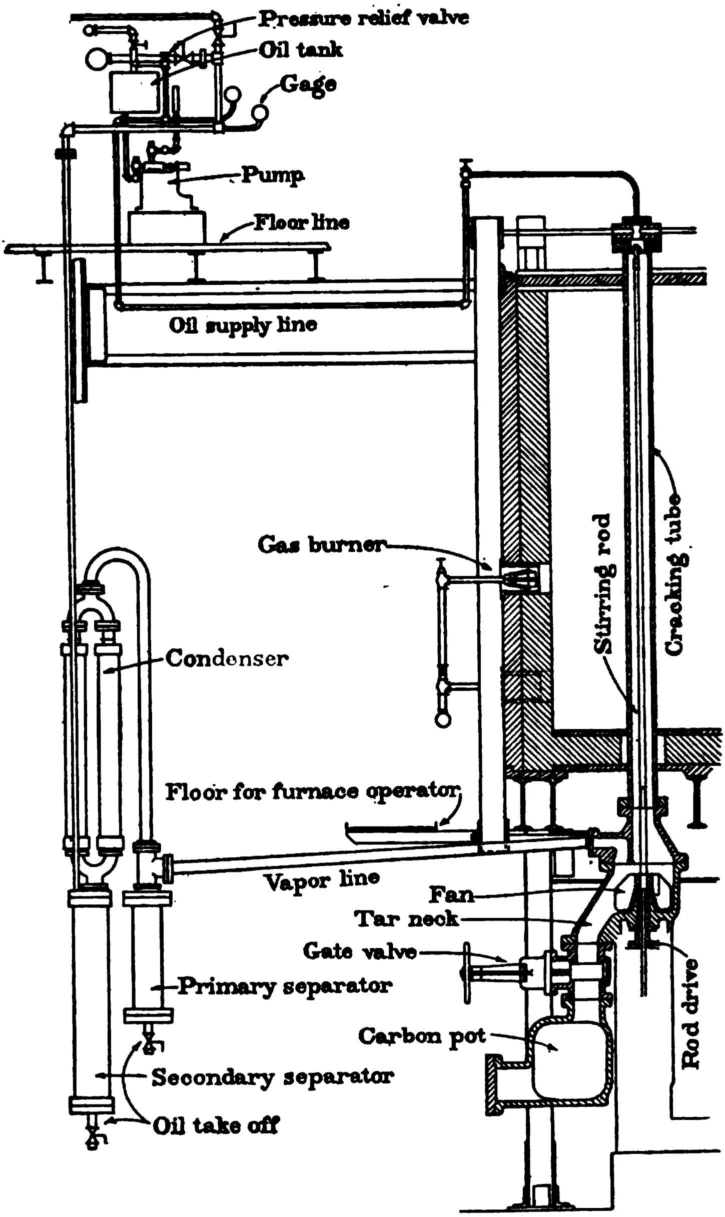
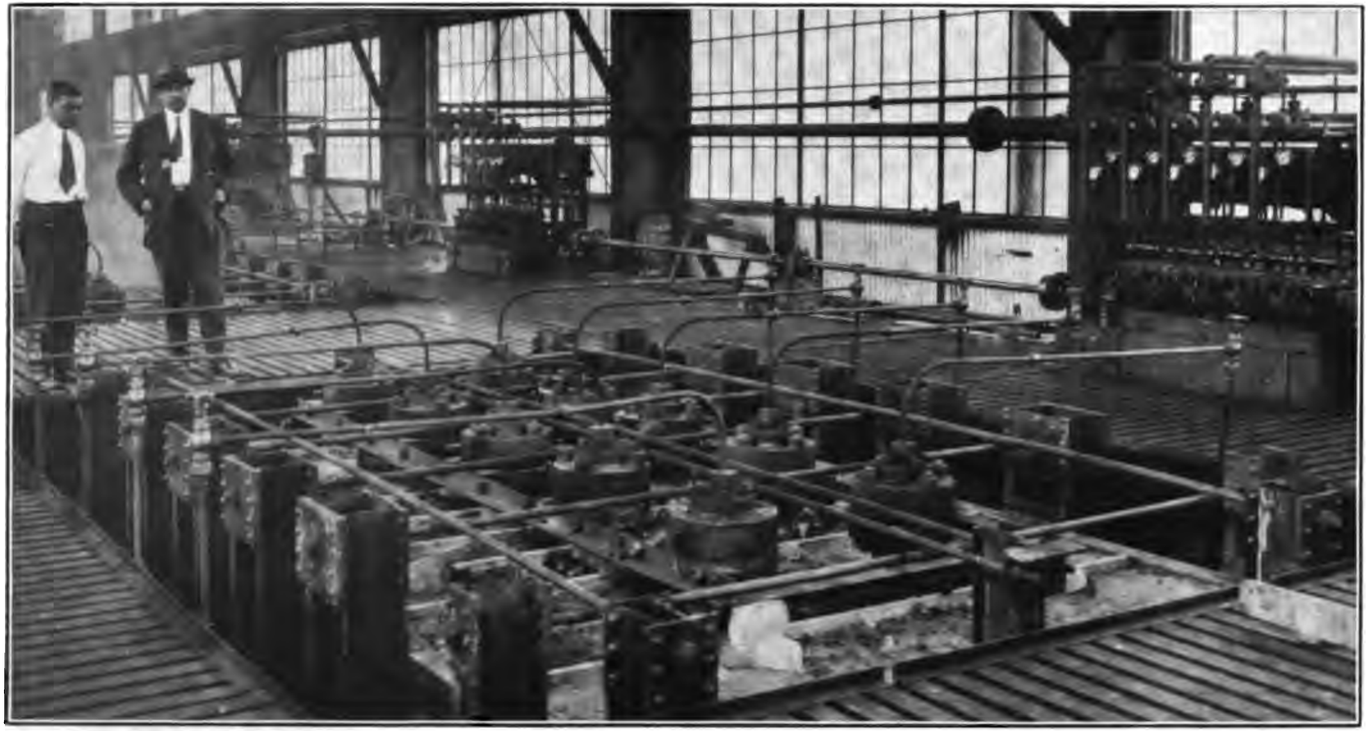
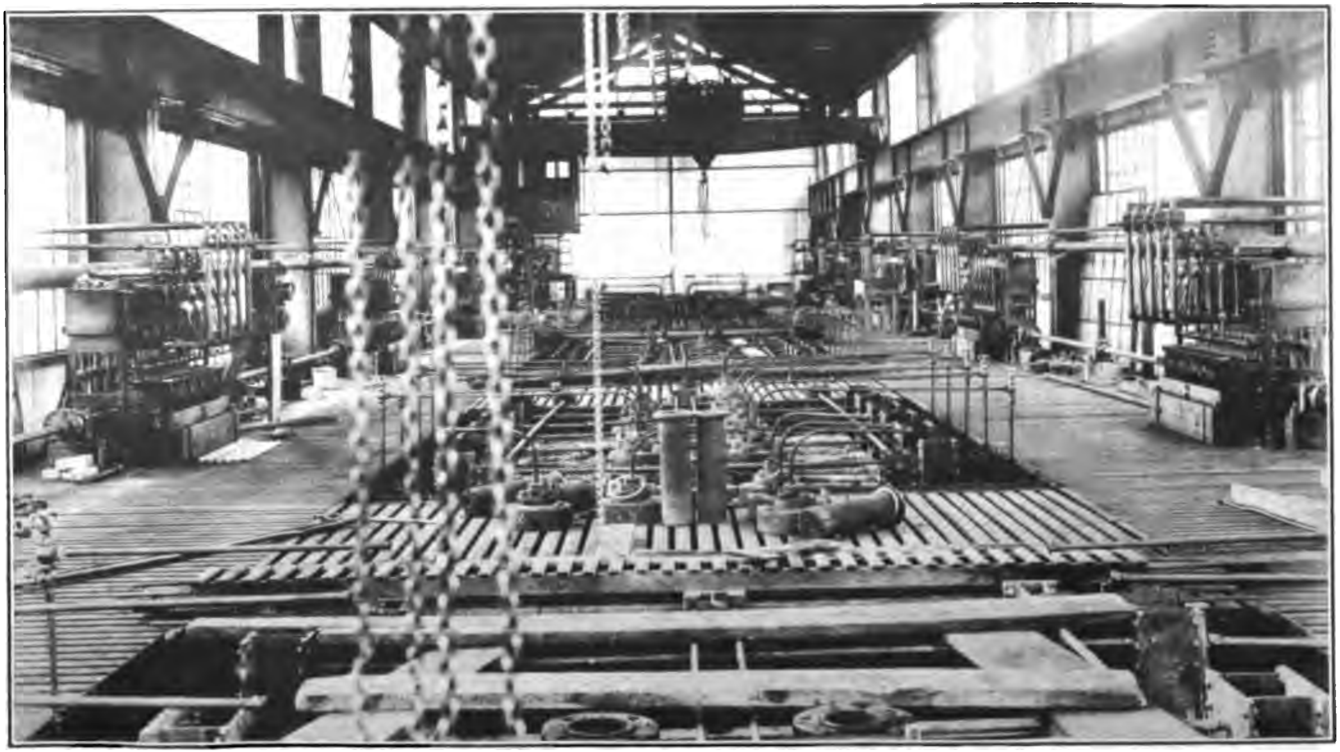


FIGURE 43.—Diagram of furnace connections.



ARRANGEMENT OF APPARATUS FOR FEEDING OIL INTO CRACKING TUBES.



GENERAL VIEW OF BALCONY FLOOR OF FURNACE BUILDING, SHOWING ARRANGEMENT OF OIL PUMPS AND PRESSURE RELEASE VALVES.

Note in the foreground short tubes with perforated cap or end to facilitate vaporization of oil. The large hole in the cap is for the stirring-rod mechanism.

header will give a uniform rate of feed into the tubes if the pressure in the tubes is automatically controlled by means of meters placed in the supply line. The installation of separate pumps was considered necessary in order to have each tube operated as a separate unit, notwithstanding the fact that 10 tubes were incased in a single furnace.

In the system as installed each tube may be regarded as a separate unit having no connection with the operation of the other tubes. This type of installation has enabled the reactions occurring in the individual tubes to be carefully studied and has given valuable data which it would be impossible to obtain if the products of all the tubes passed into a single condenser and if a common header were used for supplying oil to all of the tubes.

The individual pumps have served their purpose, but it is not recommended that such a system be used in a new installation. The piping system was so arranged that when the pressure in the tube exceeded the predetermined mark the oil delivered by the pump would be by-passed back into the feed tank.

At first oil meters for measuring the supply fed to each tube were not used, but it was found that the amounts of oil fed into the different tubes varied widely. During the first few months of operation the amount of this variation in feed was not known, as attention was being concentrated upon other problems. With the installation of a supply tank having separate compartments for the oil to be fed to each tube, accurate measurements of the amount fed in a given rate of time was made possible. As a result it was found that there was a considerable variation in the rates of feed. A typical variation is shown by the following table:

Variation in rates of feed to cracking tubes.

Tube.	Feed, gallons per hour.	Tube.	Feed, gallons per hour.
1	12	6	20
2	30	7	8.6
3	33	8	10
4	30	9	14.4
5	10	10	8

This lack of uniformity in the rate of feed was due in part to the ball seats of the pump becoming rough or dirty and air bound. Dirt in the crude oil can not well be eliminated. All oil fed into the furnace was passed through a fine-mesh sieve, but even this precaution was not sufficient to prevent the valve seat from becoming dirty. Clogging of the needle valve, with the resultant by-passing of oil, was another cause of variation in the rate of feed.

The use of meters has been adopted in order to determine accurately the amount of oil passing into the individual tubes, a meter being installed in each feed line. It has been found that through the use of meters better control and regulation of temperature can be obtained than would be otherwise possible, as the rate of feed can be accurately adjusted to conditions existing in the furnace. It is recommended that oil meters be used in any new installation, as the individual cost of the meters is small (approximately $8 each) and the benefits obtained are more than commensurate.

APPARATUS FOR REMOVING DEPOSITED CARBON.

USE OF STIRRING ROD.

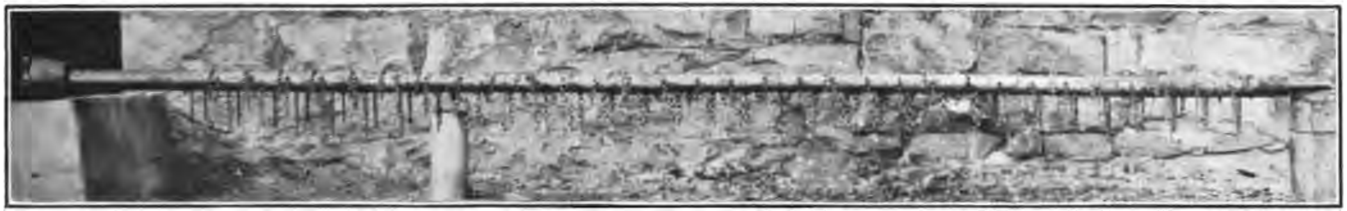
The most serious problem that was faced at the outset of operations was the removal of carbon formed on the walls of the tubes. It was realized that inability to dispose of the carbon had been the cause of the failure of other processes involving the employment of tubes. The knowledge gained from the laboratory experiments as to the percentage of carbon deposition made evident the necessity of employing some efficient mechanical means for its removal.

Carbon incrusted on the tube walls acts as an insulator and interferes seriously with the transmission of heat from the furnace to the vaporized oil; also the gradual accretion of carbon diminishes the size of the reaction chamber until the tube becomes entirely choked, which necessitates shutting down the furnace to remove the deposit. It is believed by the authors that in any process for transforming petroleum into aromatic hydrocarbons in commercial quantities by the action of heat considerable quantities of carbon will necessarily be deposited.

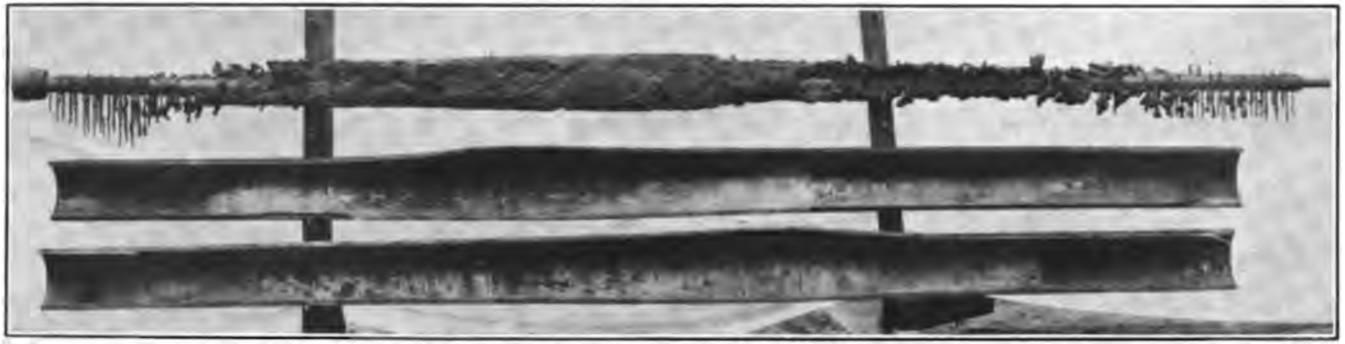
The use of cracking chambers of so large an internal diameter as 8 inches introduces another factor which makes it desirable to use stirrers. In order to heat gases in the axis of an 8-inch cylinder to any desired temperature a considerably higher temperature must be maintained at the periphery of the cylinder. One of the advantages of the use of the stirring rod is, therefore, that it makes the cracking chamber annular. Also the stirring action of the rods and chains tends to promote transverse circulation of the gases, which helps to equalize the temperatures.

CONSTRUCTION OF STIRRING RODS.

The use of stirring rods was decided on after careful weighing of the probable advantages of a number of possible devices. The construction of the rod is shown by Plate VI, *A*. It will be observed that the



A. STIRRING ROD, COMPLETE.



B. CARBON ACCUMULATION ON STIRRING ROD. LONGITUDINAL HALF SECTIONS OF TUBE, SHOWING EFFECT OF OVERHEATING.

chains are arranged spirally around the rod and are so spaced that they will scrape the entire periphery of the tube where the cracking takes place and carbon is deposited. The chains are thrown out by centrifugal force as the rod is rotated, so that the links strike the inner wall of the tube with considerable force.

The arrangement of the stirring rod in the tube, and the manner of its operation, are shown by figure 44. The lower end of the rod is detachably connected to a vertical shaft that extends upward through a stuffing box in the bottom of the tar neck and has its lower end keyed in a bevel pinion which intermeshes with the corresponding pinion on a short horizontal shaft driven by a chain from below. The entire series of carbon stirrers in a single furnace is operated by a motor-driven line shaft, which is so arranged that the stirring rods on one side of the furnace revolve in one direction while those on the opposite side rotate in the reverse direction. Clutches are attached to each short horizontal shaft, so that any number of stirring rods can be thrown out of gear while the others are being rotated.

The general arrangement of the stirring rod drive is clearly shown in Plate VII, *A*. It is apparent that if the rods are continuously rotated in a given direction no means is afforded for cleaning the rod and the chains attached thereto, so that carbon will collect until the accumulation extends to the wall of the tube. The presence of carbon on the surface of the rods and on the chain itself does not, however, interfere with the operation of the apparatus until the amount becomes sufficient to impede the movement of gases through the chamber. When this point is reached, the flow of oil into the tube must be stopped and the rod removed and cleaned.

Much more trouble was experienced with this building up of carbon on the stirring rod during the early stages of the experimental work when the crude oil was being used than has occurred in recent operations in which distillate oil was used. An idea of the character of the carbon formation upon the rods during the early stages of operations may be gained from Plate VI, *B*, showing the condition of a rod after operation in the furnace for a number of days.

After the rod has been cleaned of carbon by means of air hammers or any similar device it can be replaced in the tube and used again. With the use of the distillate oil and the decreased temperatures and pressures at which the later experiments have been conducted, the period of operation of the rod in the tube has been materially lengthened. At the outset a rod could be operated only 10 to 24 hours, when the amount of carbon deposition would compel its removal. It has been found possible to increase the length of service to anywhere from 7 to 10 days, with occasional runs of even longer duration.

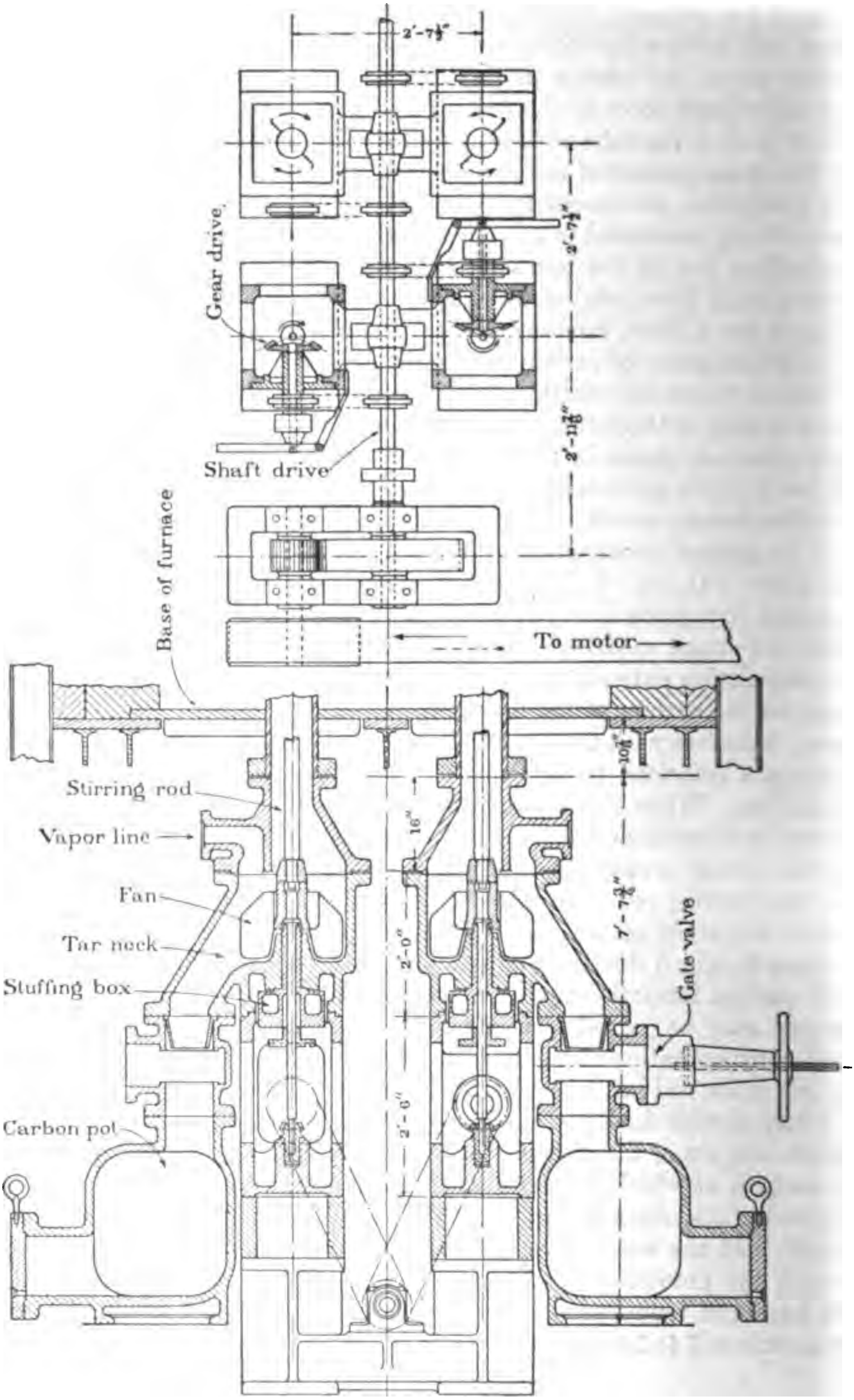
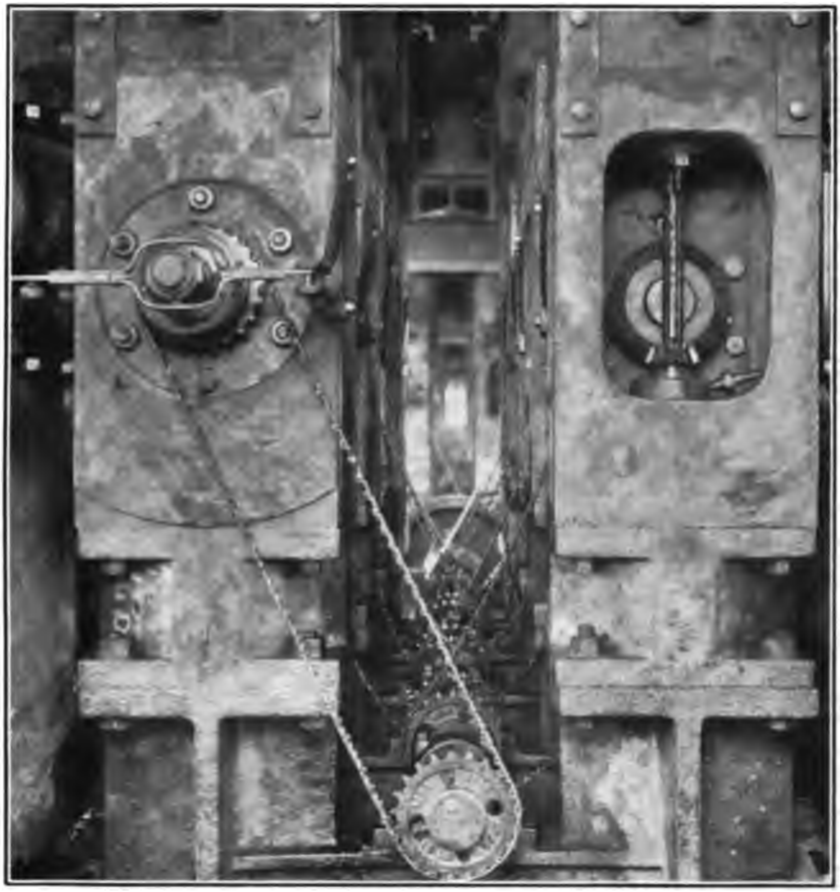
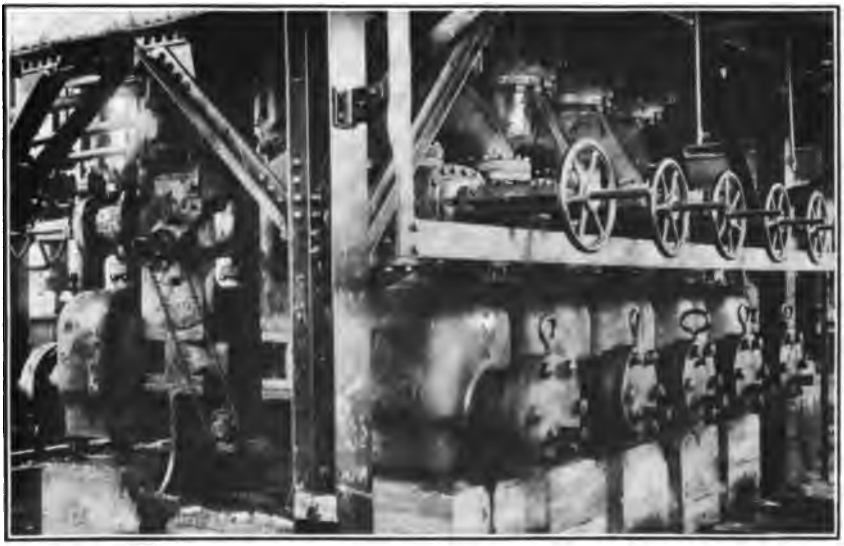


FIGURE 44.—Details of stirring rod drive and tar neck.



A. DETAILS OF STIRRING-ROD DRIVE.



B. TAR NECKS AND CARBON POTS, SHOWING ARRANGEMENT OF GATE VALVES.

OPERATION OF STIRRING RODS.

One of the chief causes of the rapid deposition of carbon on the stirring rod has been the fact that when the 10 rods were all operated at the same time such a synchronous vibration or rocking of the furnace was set up that continuous operation of all the rods was not safe. Accordingly, for a number of months, only one of the rods in a furnace was operated at a time. It was the practice to rotate an individual rod for 2 minutes, stop it, and proceed to rotate the next rod. In this way each rod was only operated 2 minutes out of every 20 during the run. The advantages of the stirrer were thus largely minimized, as the carbon had an excellent opportunity to build up during the period that rotation was suspended.

The reduction in length of rods previously mentioned and rotating of the rods on each side of the furnace in opposite directions have tended to relieve this trouble. Continued rotation of a rod in a given direction, however, does not give the full advantage possible with the chain construction, as the centrifugal force imparted by continued rotation holds each chain practically rigid, thus making it entirely similar in action to a knife or scraper.

After continued observation and experimentation had shown that unidirectional rotation of the rods was not giving satisfactory results, a reversible motor was installed for a single battery of 10 rods. This motor was so arranged that it would automatically reverse after a given number of revolutions, or it could be reversed by hand. The greater strain exerted on the rod by this reversible motion naturally required a stronger shaft than that which was previously used.

The theoretical advantage of reversible rotation is that it is best calculated to take full advantage of the chain construction of the wipers, causing them to be thrown with considerable force against each other and against the shaft of the rod itself, thus having a tendency to throw or shake off the carbon which may have formed there, and may not have been too firmly encrusted for removal in this manner. This advantage has not been manifested in operation, so that the reversible action has been abandoned, and all rods are now operated in one direction.

Various other types of mechanical carbon removers have been considered, including a plunger head to be periodically forced down through the tube. The design for such a plunger cleaner has been worked out, but the satisfactory operation of the stirring rods has caused experiments with the plunger-type cleaner to be deferred.

It is considered that the carbon troubles in the early stages of the experimental work were largely caused by the variable furnace heats which characterized operations at that time, and which, to a lesser degree, still continue. With still further improvement in the regulation of heating it is believed that wiping chains will prove absolutely satisfactory, as even under adverse conditions they operate with a considerable degree of efficiency.

The chains originally used were too light for the most satisfactory results and heavier chains are being installed. The chains are readily obtainable in the market and their attachment or removal is a simple matter. Experience with the single-tube furnaces has shown that under proper operating conditions stirring rods will last for an indefinite period. The initial cost of these rods, including the chains, is about $50 each.

In the multiple-tube furnaces, especially during the periods when excessive heating occurred so frequently, the rods occasionally manifested a tendency to soften and buckle. This difficulty, however, has been totally absent in the case of unit-tube construction.

GENERAL CONCLUSIONS CONCERNING CARBON REMOVAL.

The use of a rotating rod with wiping chains attached has demonstrated that it will keep the walls of the tubes free from carbon. With the improvements in the construction of this rod which experience has shown to be advisable, and with uniform heat control, it is believed that this type of cleaner can be used with satisfactory results. The great objection, however, to any cleaning device which is permanently retained in the cracking chamber is the fact that although it can be made to clean the walls of the tube there are no means for cleaning it. It is only a question of time, therefore, which in this case means days, before the amount of carbon built up is such as to necessitate the removal and cleaning of the device. Therefore, from a purely mechanical standpoint, a type of cleaner which is not to be permanently retained in the reaction chamber would be more satisfactory, if it cleaned the walls of the chamber equally well. It is entirely possible that a type of plunger cleaner will be found to accomplish this result and obviate the fundamental objection to the use of any intermittent cleaning device, which is the difficulty in obtaining a continuous oil feed and maintaining uniform conditions of operation. But a plunger cleaner head would be more costly to install and would necessitate detachment whenever a tube was to be removed or its condition examined. Likewise the advantages that accrue from throwing the gases against the walls of the tube would, of course, be largely lost with plungers.

CARBON POTS AND TAR NECKS.

The tar neck, so-called, is shown in figure 44. It is attached to the bottom of the tube by a flanged head, which is bolted to the corresponding flange attached to the tube. The fixed and the condensable gases, and the carbon which has been removed from the walls of the tube by the mechanical stirrer, pass downward into the tar neck or chamber.

A baffle wall or curtain in the upper part of the chamber around and below which the gases must pass, in order to enter the vapor line leading to the condenser, tends to prevent a large percentage of free carbon from being mechanically carried over with the gases into the condenser. The greater specific gravity of the carbon and pitch causes them to settle rapidly to the bottom of the chamber.

A fan is attached to the vertical shaft which engages with the stirring rod, and is continuously rotated during the progress of a run in order to keep the stirring-rod bearings from being clogged. The action of the fan has a slight tendency also to draw the gases down from the reaction chamber and into the tar neck. The gases, however, being lighter than the heavy pitch and carbon formed, will rise and pass out through the vapor line, leaving the heavy tars and carbon behind.

The lower end of this chamber is connected to what is termed the carbon pot or carbon receptacle (see Pl. VII, *B*), which is provided with a discharge spout or neck with a removable cover. Intermediate between the carbon pot and tar neck an 8-inch gate valve is placed, so that the desired pressure can be maintained in the tube proper while the carbon and heavy tars are being removed from the receptacle.

The carbon pot is set on a concrete base 2 feet 4 inches high. The receptacle itself is 2 feet 11 inches high. The interior of the pot measures about 7½ cubic feet and has a capacity equivalent to the amount of carbon formed during a run of five to six hours duration where crude oil is used. The capacity is greatly increased if a distillate is employed, as the heavy pitch and residuum of crude oil is a prolific source of carbon.

A larger carbon pot with a capacity of two to three times that of the carbon pot shown in Plate VII, *B*, has recently been installed on several of the furnaces and has made possible runs of correspondingly greater duration. The general construction of the larger pot, which is essentially the same as that of the one herein described, is shown in Plate II, *B*. In the upper part of both illustrations will be seen the flange connection with the bottom of the tube. The position of the gate valve is likewise shown.

Carbon receptacles such as those here depicted have the disadvantage of limited capacity. It has been the practice to release the pressure from the tube and to suspend operations while the carbon and pitch were being removed from the carbon receptacle. The duration of the run was fixed accordingly by the capacity of the carbon pot.

Although the gate valve is so designed as to permit the removal of carbon while oil is being cracked, it was considered desirable, while the experiments were in progress, to release the system from pressure and to ascertain that the tar neck and bottom of the tube had not become clogged with carbon or pitch.

Each carbon pot is provided with a pressure release valve, so that the gas which may have accumulated in the pot can be released before the flange head is removed. These pots were made unusually massive at the outset because of the desire to have no delay in operations due to failure of any part of the apparatus to withstand high pressure.

CONCLUSIONS AS TO CARBON POTS.

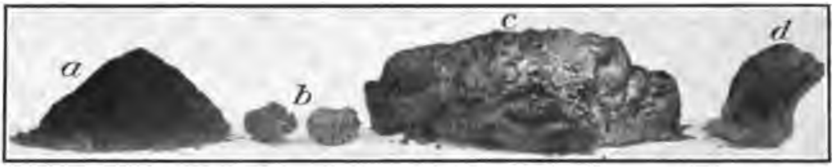
The results of the large-scale operations have shown that such massive equipment is not essential. However, this fact could not be predetermined, and it is to the credit of those responsible for the installation that safety has been the first consideration throughout the entire process of development.

The change in the character of carbon formed in the tube from a dense hard coke to a soft flaky carbon, or a heavy viscous tar, has also tended to make the use of large castings unnecessary in the future. If a clean oil is used, such as the distillate to which frequent reference has been made, there is every reason to believe that in place of individual carbon receptacles the tar and carbon can be run from the tar neck direct into a common header and discharged from there into a suitable receiving tank. In fact, provision to this end is now being made at the development plant. If the residuum of recovered oil above the 175° C. cut, or the liquid product from the condenser, is pumped through a suitable opening in the tar neck, there should be no difficulty in causing the carbon and pitch to flow freely through the header. The oil so used can be recovered readily.

The character of the carbon formed in the process is illustrated in Plate VIII, *A*. The soft flaky carbon is the kind that is formed when proper tube temperatures are maintained throughout a run on Oklahoma crude oil. This soft carbon is usually accompanied by a heavy tar sludge, which is not generally as viscous as that shown in the illustration. Coke is formed when excessive furnace heating occurs. With careful regulation of burners very little of this coke will be produced. The carbon constitutes a valuable by-product and is adapted to a wide variety of industrial uses. It is worth from 1 cent per pound upward, depending on its condition and relative freedom from metallic oxides.

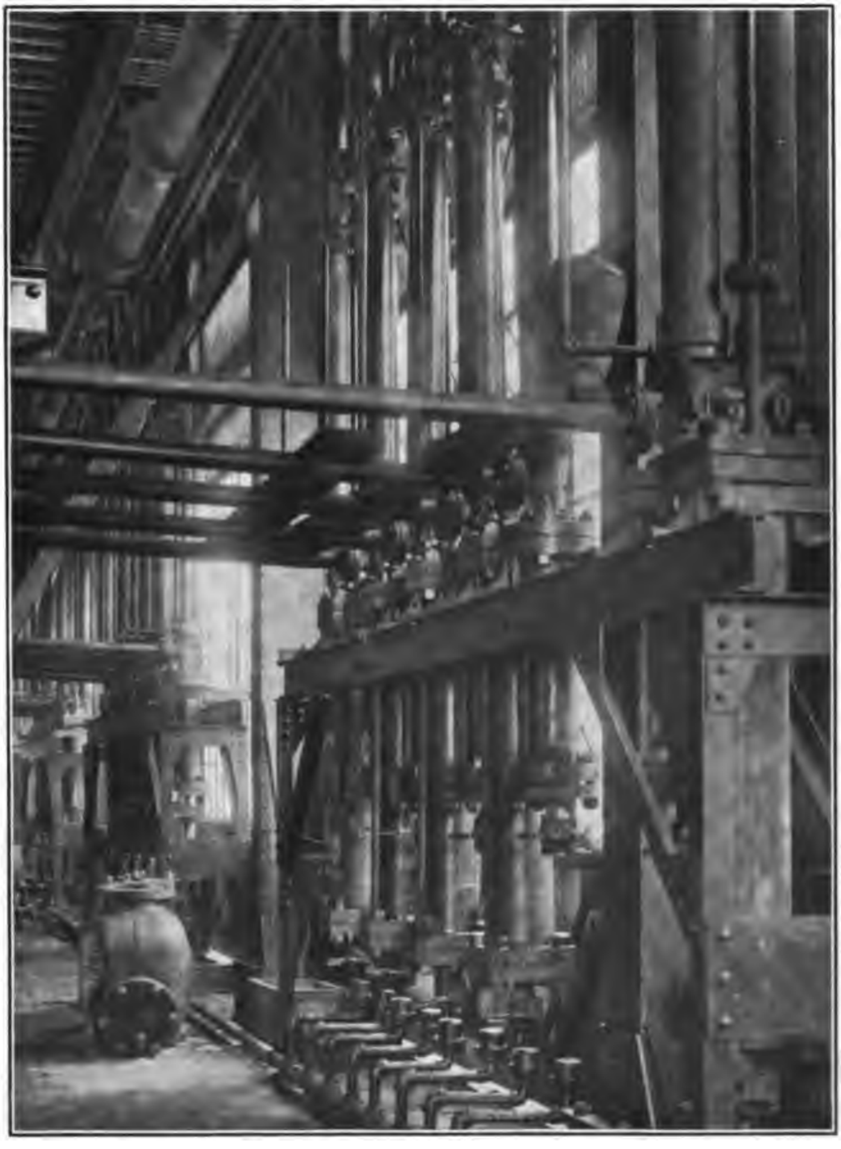
CONDENSERS.

At the outset of the experiments it was felt desirable, as previously mentioned, to have each tube operated as a separate unit, thus making it a complete system in itself. This necessitated the use of an



A. CARBON FORMED IN OPERATION OF BENZENE-TOLUENE PROCESS.

a, Soft powdery carbon; *b*, light coke; *c*, heavy resinous tar; *d*, dense coke. (Such coke gave trouble in the early stages of developing the process.)



B. ARRANGEMENT OF CONDENSERS FOR MULTIPLE-TUBE FURNACES.

individual condenser for each tube. If this plan had not been followed a study of the conversion results in the separate tubes of a single furnace would have been out of the question.

In all new processes a great deal of the equipment considered necessary at the outset can be dispensed with subsequently. This is the case with the individual condensers. Experience has shown that the gaseous products of the reaction can be discharged through the vapor line connected with each tube into a common header, and thence be passed to a multiple-coil condenser such as is in common use in oil refineries. It would have been neither safe nor wise to have adopted this system at the beginning, and consequently the expense involved in the installation of 60 separate condensers was made necessary. The fact that the condensers were in place has led to their continuous use. It is now proposed, as has been stated, to pass the gases through these condensers so as to get such cooling effect as the condensers will give, and then through a common header into a multiple-coil condenser. The fixed gases and vapors leaving the condenser will be passed through a gas scrubber, after which the gases may be released into the open air or be carried to a gas holder, whence they can be drawn upon as required for fuel in the furnaces.

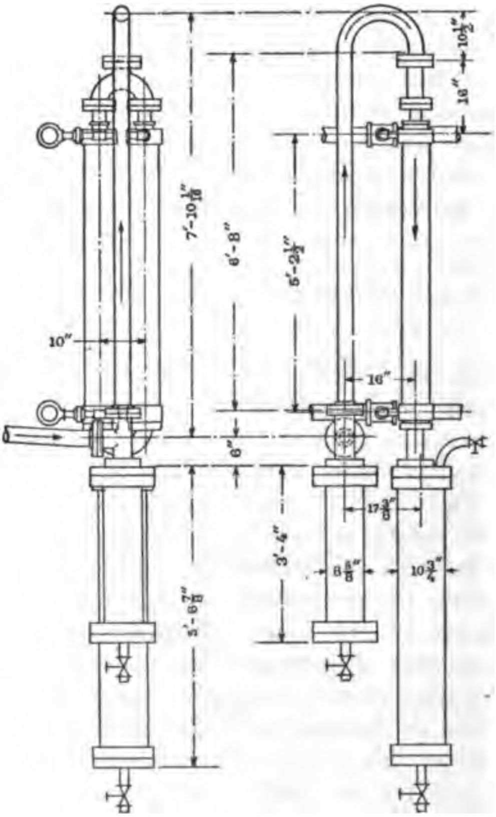


FIGURE 45.—Details of condenser construction.

DETAILS OF CONDENSER CONSTRUCTION.

The arrangement of these condensers in the furnace building is shown by Plate VIII, *B*. The details of construction of the condensers are given in figure 45.

The gases are carried to the condenser through a 3-inch pipe some 12 feet in length. A receptacle termed the primary separator is connected with the vapor line, in which the more readily condensable liquids are collected. The gases which remain uncondensed at this point pass upward through a 3-inch U-shaped iron pipe into a twin water-cooled condenser consisting of 3-inch copper pipes incased in iron pipes through which water is continually circulated. The liquids which are condensed pass by gravity into what is called the secondary separator. The fixed and uncondensed gases are discharged into a gas header on the upper floor of the furnace building by means of a 1½-inch pipe whenever the pressure on the system exceeds the predetermined pressure. The primary separator is 8⅝ inches in diameter by 3 feet 4 inches in height. The secondary separator is 10¾ inches in diameter by 5 feet 6¾ inches in height. These separators hold as much liquid as will be condensed during a run of the duration permitted by the capacity of the carbon receptacle. The twin water-cooled condensers are approximately 7 feet 10 inches in height and have an internal-wall area of approximately 10 square feet.

FATAL ACCIDENT DUE TO TAPPING CONDENSERS WHILE UNDER PRESSURE.

In the operation of the plant it has been the practice to keep the liquid products from the various tubes separate until after suitable laboratory tests and determinations were made, after which the combined products were discharged into a header and pumped to the storage tank to await distillation. In order to tap the condensers with safety, in view of the pressure on the system, ranging from 150 pounds to 250 pounds per square inch, it has been the custom to release the pressure before opening the cadmium valves for the discharge of the liquid. Explicit instructions were given from the beginning of operations for the observance of this precaution. The one time when orders were not followed and the separators tapped while the system was under pressure resulted in an accident fatal to five of the company's employees. The uncondensed gases were expelled under high pressure from the condenser, and immediately caught fire, thus igniting the oil-spattered clothing of the men. While this accident was most deplorable, such accidents may happen in any industrial establishment when prescribed safety precautions are not followed, and is in no wise to be attributed to any inherent danger in the use of the process itself.

INADEQUACY OF CONDENSER INSTALLATION.

The inefficiency of the condensers, which led to the installation of a large-size condenser and gas scrubber, is shown by the amount of aromatic hydrocarbons in the oil immediately on leaving the condenser and the amount in the same oil after it had been exposed to the air in open barrels, in which it was customary to store it temporarily. It was found that between 30 and 50 per cent of the low-boiling aromatic hydrocarbons formed in the tube had evaporated while the oil was allowed to stand in contact with the air. The significance of this loss has been shown in the discussion of the data contained in Table 57. Some liquid was mechanically carried over by the gases and did not condense in the individual condensers.

The volume of gas formed in the cracking process is so great that a very considerable percentage of both light and heavy oils is mechanically carried over and does not yield readily to condensation. Traps were placed in the gas line, into which the uncondensed gases were discharged, and analyses have been made for a number of different runs. It was found on distillation from the traps that 50 to 60 per cent boiled over below 175° C., in which cut the benzene content ranged from 5 to 21 per cent and the toluene content from 8 to 25 per cent.

In the effort to further determine the amount of products which had been lost in this manner, the gas issuing from the header was passed through a scrubber with the result that large percentages of benzene and toluene were recovered. These losses, it will be observed, are in addition to the losses by evaporation from the recovered oil taken from the separators.

A table showing the percentages of benzene and toluene in the so-called header oil is inserted for the purpose of showing the extent of the losses which resulted during the time covered by this publication and of indicating clearly the need of adequate condensing and scrubbing apparatus.

Benzene and toluene in header oil.

Percentage of oil distilling up to 175° C.	Specific gravity of distillation cut.	Percentage of benzene in header oil cut to 175° C.	Percentage of toluene in header oil cut to 175° C.
72	0.844	8.9	12.0
63	.860	21.0	25.0
66	.890	8.9	13.2
58	.838	11.0	16.0
49	.842	5.4	8.8
49	.868	7.8	10.8
60	.865	10.8	14.4
53	.870	9.0	12.2

It will hardly be credited, perhaps, that operations were permitted to continue for such a length of time without making suitable provision for the complete condensation and separation of the liquid products from the permanent gases. The chief factor in this situation was the continued desire to study the results accomplished by the individual tubes, which would not have been possible had the general condensing system been in use. The continued variation in furnace heats made this study necessary in order that the furnace operators might be informed as to the temperature required for the most satisfactory general results.

CONCLUSIONS REGARDING CONDENSERS.

In any new installation for the employment of this process, provision should be made for a condenser of the same type as that used in oil refineries. With the benzene-toluene process, the condensation problem is, however, markedly different from that of the refinery where all the vapors coming from the still are condensible, as in making benzene and toluene considerable volumes of fixed or noncondensible gases are generated. In general a condensing area of approximately 2½ feet for each barrel of original oil used per day will be found satisfactory if a gas scrubber is operated in connection therewith. This gas scrubber can be modeled after the benzene scrubbing towers in by-product coke-oven plants, or may consist merely of a large tank filled with a heavy oil through which the gases are bubbled. This tank should be so inclosed that the fixed gases can be conducted to a gas holder for the purpose of supplying fuel for the furnaces, if desired. In by-product coke-oven practice the introduction of back pressure by forcing gas through a body of oil would seriously affect the coking products, whereas in the process herein described there is sufficient direct pressure to work against any desired head of oil.

GENERAL DESCRIPTION OF CRACKING SYSTEM IN COMMERCIAL PLANT.

The cracking system, both in the benzene-toluene and gasoline processes, is a closed one. Pressure is maintained throughout the entire system while operations are being carried on. The component parts of the system are shown in figure 43, in which the various parts are labeled.

The oil is delivered by the pump through the feed line passing under the upper floor, and is carried into the tops of the tubes at a predetermined rate of feed. In the benzene-toluene process the rate of feed is approximately 15 gallons per tube per hour. This rate would be more than doubled in the gasoline process for tubes of the same size. Only a very small quantity of oil is present in the tube in a gaseous form in a given period of time, as a rate of 15 gallons per hour is equivalent to the delivery of only 1 quart of oil per minute. If the oil is preheated, a much higher rate of feed can be attained in both processes.

The oil, upon entering the top of the tube, is spread out over the surface of the basket, which consists of a cast-iron pipe with a perforated top. In the laboratory experiments a basket filled with steel balls was used. These balls were also used in the furnace containing a battery of ten 4½-inch tubes. It was found, however, that the same results could be obtained by dispensing with the obstructing material and using a spreader of the type mentioned.

The pumps are so set as to deliver the oil under a pressure greater than that in the tubes, so that no difficulty is experienced in causing the oil to flow into the tubes regardless of the pressure employed. The hydrocarbon material fed in is flashed or instantaneously transformed into a gaseous condition and passes downward through what is termed the "cracking zone" of the tube proper, where a temperature is maintained higher than that in the gasification zone. It is in this part of the container that the heavy hydrocarbons are broken up under the influence of temperature and pressure and in the presence of hydrogen and fixed hydrocarbon gases. Most of the cracking occurs in this zone, although there may be some cracking immediately below the point of gasification. Although not absolutely necessary, it has been found preferable to effect gasification at a point above the cracking zone.

The condensible gases, which are the desired conversion products, pass out of the tube through the tar neck and enter the vapor line leading to the condenser. The gases which remain uncondensed ascend through the gas line connected with the top of the secondary separator and are discharged through pressure release valves either into the open air or into the gas header. The release valve discharging into the open air is used so that in case of any difficulty arising the pressure can be released in the minimum space of time.

Plate IX shows the laboratory at the development plant, including the distillation apparatus used in analyzing cracked oil.

OILS SUITABLE FOR USE IN THE PROCESS.

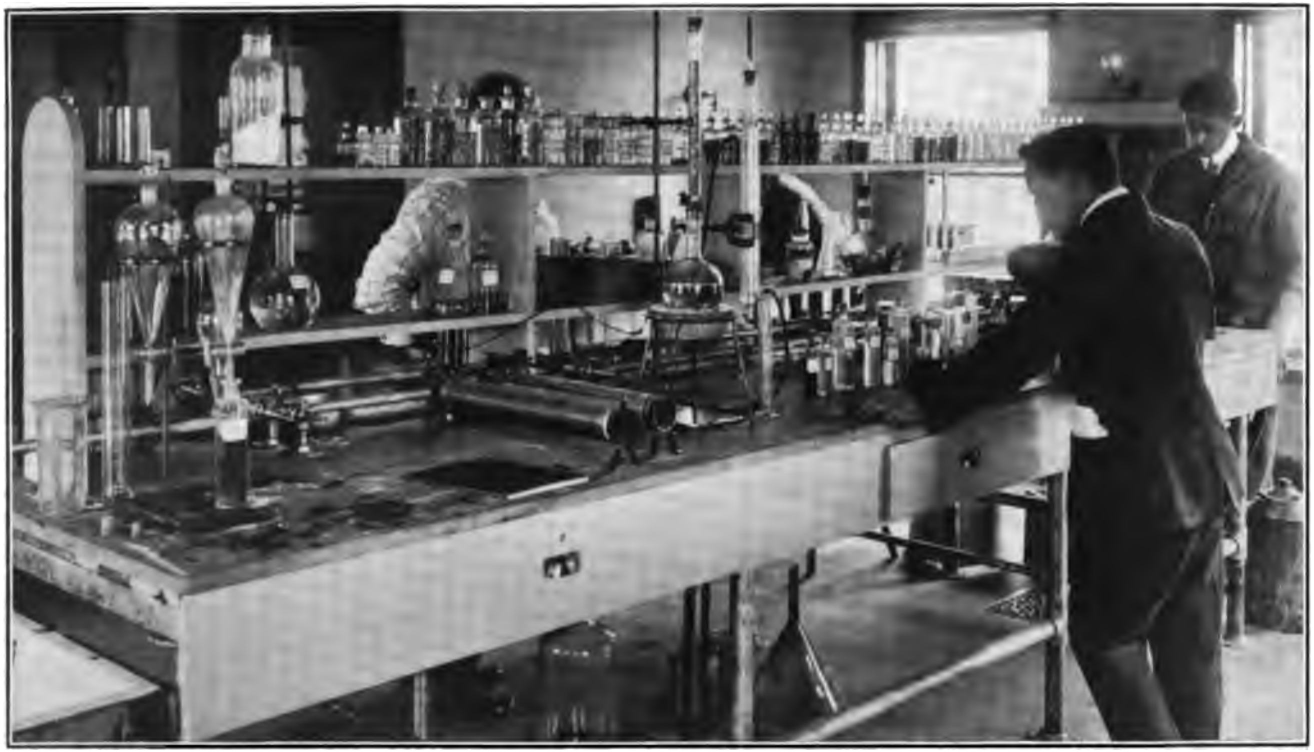
In the course of the experiments a large number of petroleum oils have been used, ranging in character from crude oils to gas oils and cylinder stocks. It was found, as was the case in the laboratory experiments, that the reactions desired were in no wise dependent on the character of oil used and that the conditions of operation were of more consequence than was the physical or chemical nature of the initial material.

The process, however, is not limited to the use of petroleum products, but can be efficiently operated on liquid hydrocarbon material derived from any source. Solvent naphthas, and light oil distillates from water-gas tars and coal tar have been successfully used. These light oils and solvent naphthas are extremely favorable for use in the process as yields two to three times larger than those obtainable from petroleum products can be had in a single operation. The petroleum oils, however, give a wider range of by-products. This fact, together with their greater availability, as well as lower cost, probably renders them most suitable for use. Wherever supplies of the distillates from coal or water-gas tar are available in quantity, they are a most prolific source of benzene and toluene. If such distillates were procurable in the open market their use would be highly profitable at a time like the present (1915), when both benzene and toluene are commanding heretofore unheard-of prices.

The great advantage in the use of light-oil distillates from coal and water-gas tars, aside from the greater yields obtainable, is the fact that diminished pressure can be used. Vacuum or atmospheric pressure has been shown to give results as favorable with these oils as higher pressures. This is evidenced by a study of Table 64 following, which shows the results of solvent naphtha tests in a 6-inch by 9-foot tube. The values reported here are on the basis of the percentage change in the distillation cuts and not on the basis of the total percentage of benzene and toluene formed.

These solvent naphtha runs were made primarily with a view to determining the percentage of toluene that could be obtained, because of the greater demand for that product. Although the results obtained are not of a uniform character, owing to variations in rate of feed, temperature, and pressure, the effect of changing temperature and pressure is clearly brought out by a study of the differences in the cuts with the changes in these control factors. At 600° C. and atmospheric pressure and a rate of feed of 12 gallons per hour a 10 per cent increase in the cut between 100° and 120° C. was obtained, whereas with the same temperature and approximately the same rate of feed and a pressure of 250 pounds the percentage of change was more than doubled. At the same time the percentage of oil recovered dropped from 92 per cent to 58 per cent. This loss counterbalances the increase in yield of toluene. At a temperature of 750° C. it will be noted that in the toluene cut (100°–120° C.) the percentage of increase shows a decided falling off, whereas the cut up to 100° C. increased, indicating the undesirability of the higher temperature for maximum conversion into toluene.

These experiments clearly establish the fact that solvent naphtha is a desirable material for converting into toluene on account of the high percentage of xylene and cymene, which readily degrade into toluene. Table 63, showing the results of the experiments, follows.



LABORATORY AT DEVELOPMENT PLANT, SHOWING TYPE OF DISTILLATION APPARATUS FOR ANALYZING CRACKED OIL.

TABLE 63.—*Benzene and toluene formed from cracking solvent naphtha in single-tube furnace (tube 6 inches in diameter and 9 feet long) at varying pressures, temperatures, and rates of oil feed.*[a]

Number of runs.	Pressure, pounds per square inch.	Temperature, °C.	Rate of oil feed, gallons per hour.	Percentage of oil recovered.	Results of distillation on basis of original oil.				
					Oil recovered at 0° to 100° C., per cent.	Oil recovered at 100° to 120° C.		Oil recovered at 120° to 150° C.	
						Per cent.	Specific gravity.	Per cent.	Specific gravity.
1	Atmospheric.	550	13	100	2.2	4.7	....	68.7	0.860
1	...do...	550	20	92	1.8	4.6	....	67.5	.856
1	...do...	600	9	75	.7	14.2	0.880	41.3	.877
1	...do...	600	12	100	2.5	10.0	.859	59.0	.858
1	...do...	600	18	92	1.4	12.9	.869	60.0	.869
4	...do...	650	13	80	1.6	10.4	.867	49.2	.870
1	...do...	700	10	83	2.9	9.9	.863	52.0	.865
2	...do...	700	13	69	2.1	14.9	.863	36.5	.862
1	...do...	750	14	21	1.4	2.6	.868	7.5	.866
1	250	550	7	83	1.8	6.6	.864	58.5	.876
1	250	550	14	83	1.6	8.7	.866	54.5	.868
1	250	600	14	58	2.4	22.4	.880	21.0	.877
1	250	600	20	67	.5	3.2	....	46.5	.876
1	250	650	8	46	1.0	17.3	.873	17.7	.880
1	250	650	14	67	4.5	18.1	.866	28.5	.866
1	250	650	17	50	1.5	12.5	.864	23.5	.864
1	250	650	21	58	3.5	12.6	.874	26.5	.868
1	250	700	24	96	....	....	....	23.0	.865

[a] Results low, due to insufficient condensation.

DISTILLATION ANALYSIS OF SOLVENT NAPHTHA USED.

[Specific gravity, 0.868.]

Temperature of cut.	Proportion of original oil distilled.
° *C.*	*Per cent.*
100	Few drops.
120	3.75
150	76.50
160	12.00
170	5.50
([a])	2.25

[a] Loss.

The residue of cracked petroleum oil—that is to say, the fraction boiling above the toluene cut—has been shown to be highly suitable as a further source of benzene and toluene because of its xylene and cymene content. The results of experiments with the residue of cracked Oklahoma crude oil from which the low-boiling aromatic fraction has been removed are given in Table 64

In considering the yields reported in Table 64 it should be remembered that the absence of adequate condensation facilities has made the yields lower than would otherwise be obtained for reasons which have heretofore been pointed out. The data, however, agree with those previously obtained, and clearly show that the residuum of the cracked oil is favorable for reconversion.

TABLE 64.—*Benzene and toluene formed in a single-tube furnace (tube 8 inches in diameter and 10 feet long) from cracking the residue above 175° C. of an oil previously cracked at varying pressures and rates of oil feed.*[a]

Number of runs.	Temperature.	Pressure per square inch.	Rate of oil feed per hour.	Oil recovered.	Specific gravity of recovered oil.	Benzene in recovered oil.	Toluene in recovered oil.	Total benzene and toluene in recovered oil.	Benzene formed, on basis of original oil.	Toluene formed, on basis of original oil.	Total benzene and toluene, on basis of original oil.
	°C.	*Lb.*	*Gal.*	*Per ct.*		*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
1	600	Atmospheric.	14	79	1.035	3.2	3.2	6.4	2.5	2.5	5.0
1	700	...do..	15	70	1.052	3.3	3.0	6.3	2.3	2.1	4.4
3	650	50	15	49	1.093	9.0	3.7	12.7	4.5	1.8	6.3
1	650	25	15	39	1.065	15.9	7.6	23.5	6.3	3.0	9.3
1	700	25	21	54	1.100	13.0	3.5	16.5	7.1	1.9	9.0
1	650	100	21.2	27	1.048	13.0	5.2	18.2	3.5	1.4	4.9
1	700	100	14.0	21	1.014	14.6	7.5	22.1	3.1	1.6	4.7
1	700	100	22.5	31	1.058	11.5	4.9	16.4	3.6	1.5	5.1
1	700	100	28.8	39	1.060	8.1	3.8	11.9	3.2	1.5	4.7
1	575	150	14	62½	1.034	4.3	3.8	8.1	2.7	2.4	5.1
1	600	150	14	44	1.062	8.3	4.8	13.1	3.6	2.1	5.7
1	650	150	16.5	48	1.042	8.2	5.2	13.4	4.0	2.5	6.5
1	600	200	15.5	41	.989	7.0	6.0	13.0	2.9	2.5	5.4

[a] Results low, due to insufficient condensation.

CONCLUSIONS AS TO OILS.

The experiments have clearly indicated that the vapor-phase cracking process is not limited to any particular type of oil, and that an asphaltic oil, a paraffin-base oil, oil having a paraffin and asphaltic base, and certain oils obtainable from the destructive distillation of coal, are all suitable for use in the process.

RELATION OF COMBUSTION SPACE TO TEMPERATURE

As has already been stated, the furnaces used for the cracking of the hydrocarbons in a gaseous condition are heated by natural gas. Excellent results were obtained in the single-tube furnaces, but it has been found rather difficult to maintain uniform heats for the 10 tubes in a single combustion chamber. A careful checking of the conversion results accomplished in the various tubes has shown that a consistently higher temperature is maintained in the tubes nearest the stacks, and that the tubes farthest from the stacks show the smallest amount of conversion.

The combustion chamber in the multiple-tube furnace contains approximately 1,100 cubic feet of space or an average of 110 cubic feet per tube. This combustion volume seems to be insufficient for the best results under the conditions of operation which have existed during the period covered by this report. It is believed, however, that much better results could be obtained with this volume of furnace per tube if it were to be combined with an efficient outer combustion chamber

METHOD OF CONTROLLING TEMPERATURE.

In the operation of the plant a laborer, termed a heater, is employed for each furnace of 10 tubes. This man has charge of the 22 burners required to heat a single furnace, and is engaged constantly in the regulation of the burners in order to maintain the required temperature. This regulation of heat is controlled by optical determination of the temperature, as judged by the condition of the tube and brickwork in the interior of the furnace. Pyrometers have also been used, but have given results of varying character. The optical readings, when made by operators with sufficient experience, have permitted a degree of control more satisfactory than that obtainable through dependence on pyrometers alone. It has been found practically impossible to maintain an insulation which would permit continuous use, as all of the various insulating materials used, such as asbestos, fire clay, fire brick, or a combination of the three, have rapidly given way under the conditions existing in the chamber. Automatic regulation of temperature by means of a thermostat, it is believed, can be ultimately obtained.

The short life of the pyrometers, notwithstanding attempts to protect them by incasing in a suitable rod, have constituted a considerable item of expense, and experience has demonstrated the inadvisability of depending on pyrometers alone for temperature control. It has been found that when eye readings are taken by means of a cold iron pipe inserted through one of the peepholes in the furnace shell, the contrast in color between the cold pipe and the glowing interior of the furnace has enabled the heater to accurately gage the temperature. It has become evident, therefore, that eye control, supplemented by pyrometric readings, the pyrometer to be periodically inserted in the combustion chamber, is the most advisable means for obtaining uniform results unless an efficient thermostat be used. The eye quickly becomes trained and permits the approximation of temperature with remarkable accuracy.

The wide use of gas-heated furnaces in various industries, and the high degree of uniformity in heat which has been obtained by long experience indicates that the heating problem should not in the future be as serious a difficulty as it has proved to be with the 10-tube furnaces in the experiments reported herein.

Control of temperature in the single-tube furnaces, with the exception of the 8-inch by 14-foot furnace, was easy and there is no question but that the single-tube furnace lends itself much more readily to accurate control than the larger installation.

The influence of temperature on the character of the reaction has already received detailed comment in connection with the discussion of theoretical considerations in the first part of this paper.

METHOD OF CONTROLLING PRESSURE.

Pressure in the cracking system is maintained by controlling the discharge of the gases formed by the decomposition of the hydrocarbons fed into the tube. In the operation of the process at the development plant a laborer is employed for each unit of five tubes in order to regulate the pressure. Regulation is accomplished by opening a valve and permitting the gases to escape into a gas main or header until the pressure in the individual tube has been sufficiently lowered to keep it at the point required. For a furnace of 10 tubes, two men are employed to control pressure conditions.

There is no reason why in new subsequent installations the services of these men can not be dispensed with by using automatic valves which can be set to release at the desired pressure. It was undesirable to do this in the course of the operations described in this bulletin for the reason that absolute control of operations could be better attained by hand regulation. It will be realized that in the first stages of the development of any process, labor costs are always high until after procedure has been so far simplified as to permit the substitution of mechanical control for manual labor. There seems to be no reason why a substantial reduction in operating expenses can not be effected by mechanical regulation of the pressure.

At the outset of the experiments it was considered desirable to obtain as large quantities of benzene as possible. Therefore, a uniform pressure of 250 pounds per square inch, which had been shown to give the best yields, was adopted. After a considerable period of time it became of more consequence to increase the percentage of toluene formed, for which a lower pressure is more favorable; consequently a number of experiments were conducted which established the fact that for the distillate oil a uniform pressure of 150 pounds per square inch is the most satisfactory from a commercial standpoint. A reasonable degree of conversion is obtained at this pressure which, coupled with the improvement along mechanical lines, makes 150 pounds the most favorable pressure when toluene is the chief product desired.

CARBON FORMATION.

As was stated in the discussion of temperature, carbon difficulties increase when heat is not uniformly regulated. The experiments again indicated the already well-known fact that the percentage of carbon formed increases with temperature.

Although the temperatures (600° to 750° C.) favorable for formation of low-boiling aromatic hydrocarbons are sufficiently high to make unavoidable the formation of a considerable percentage of carbon, the amount of carbon formed is also dependent to a large extent on the character of the original oil. The Oklahoma crude oil, which was used in the first runs formed on the average 1 to 1½ pounds of carbon per gallon of oil. The distillate which was substituted formed one-fourth to one-half pound per gallon. With both types of oil, when temperatures and rate of feed are in proper relationship, the carbon is soft and flaky in character and of fine texture, or else is in the form of a tar. The soft carbon resembles lampblack to some extent, and is a useful by-product. When the carbon is in this condition, the use of stirring rods will efficiently prevent incrustation of carbon on the walls of the tube.

It is only when the heat becomes excessive that a layer of coke accumulates on the inner wall of the tube. When dense, hard, coke forms, it is difficult to remove. As has been previously suggested, the use of heavier chains than those employed on the stirring rods in the experiments reported in this bulletin might noticeably increase efficiency in removing carbon.

The nature of the carbon formed when excessive heats are permitted is shown in Plate VI, *B*. It will be noted in the illustration that the carbon is built up on the stirring rod to the full diameter of the tube, completely choking it and preventing the passage of gases generated in the upper part of the chamber. Considerable difficulty was experienced with this type of coke, or carbon, in the early stages of the experimental work. Its formation was due to three causes: First, to the higher temperatures, which were used in order to obtain the maximum conversion possible in a single operation; second, to the inexperience of the heaters, which resulted in excessive heats; and third, to the greater carbon deposition incident to the use of Oklahoma crude oil as original material.

With the change in the character of the oil used, and a somewhat improved heating control, practically no dense coke has been formed on the walls of the tube, and the carbon has been removed either in the form of a tar, or else in the form of the soft flaky carbon previously mentioned. More recently (December, 1915) the carbon has been removed from the carbon pots in the form of a heavy oil, which has been mixed with the product from the condensers. This fact alone will indicate to what extent the carbon difficulties have been overcome.

The presence of carbon in the reaction chamber is a disadvantage from the mechanical side alone. From various references in the appended bibliography it can be discovered that carbon is one of the best catalysts for the formation of benzene and toluene. The mechanical difficulties from the deposition of coke arise not alone from the fact that it chokes the passage of the gases, but also hinders heat conductivity. The formation of coke on the inner wall of the tube will cut down the conductivity several hundred per cent, so that a much higher temperature is required to bring the temperature within the tube up to the desired point.

CONCLUSIONS REGARDING CARBON FORMATION.

It is evident from the development of the process that the formation of carbon in the cracking tube is not an obstacle to the operation of the process when control conditions are in proper relationship. Too low a rate of feed at a moderately high temperature will cause higher percentages of carbon to form. Excessive temperatures and pressures will likewise promote excessive carbon formation, but when these factors are in their proper relationship the character of the carbon is such that little or no difficulty is encountered in its removal.

MATERIAL USED IN TUBES.

Double extra-heavy lap-welded tubes with walls five-eighths inch in thickness have been used for the cracking chambers. These tubes come in stock lengths of from 20 to 23 feet, so that a single length may be cut into two cracking tubes. The initial cost of these tubes in the stock sizes is approximately $85 so that a cracking tube will cost approximately $42.50.

BULGING OF TUBES FROM OVERHEATING.

Lap-welded tubes have not proved entirely satisfactory. Defects developed from time to time in the seams, under the high furnace heats, so that the tubes would fail during a run. In spite of this tendency, however, there has never been a case of outright bursting in all the eight months of operation. The tubes contain only gaseous hydrocarbons, and the expansion incident to their combustion on exposure to air has not been sufficient to prove dangerous. In general, the only indication given of a break in a tube is the falling off in pressure which results. During the first few months of operation the tubes frequently did not average more than 10 days' heating before they started to bulge. A typical case of this sort of tube trouble is shown by the photograph of a longitudinal section of a displaced tube (see Plate VI, *B*).

Most of the tubes which were displaced manifested the effect of overheating. Some oxidation had occurred, but in few if any tubes had it progressed to such an extent as to indicate that it was the cause of the tube yielding. The local superheating for the most part ranged over that part of the tube which was incased in the fire-brick casement or chimney previously described, although in some instances the bulge had started at a point above the top of the checkerwork. It seemed that as soon as excessive heating had caused a tube to soften in some place, bulging resulted immediately through the effect of internal pressure. This bulging or flow once started continued until the walls had become so thin that they ruptured under the pressure. In a number of cases the metal in the tubes at the point of rupture had been thinned down to a knife-like edge.

LIFE OF TUBES.

The short life of the tubes in the multiple-tube furnaces during the earlier stages of development is in striking contrast to their life in the single-tube furnaces, where individual tubes were used for months without removal. This fact indicates the much better temperature regulation attained. Since the large furnaces have been operated at a temperature ranging from 625° to 650° C. and a pressure of 150 pounds per square inch, as compared with a temperature of 700° C. and a pressure of 250 pounds per square inch in previous runs, the life of the tubes has been greatly prolonged and tube trouble has practically disappeared, even with the lap-welded tubes.

A small number of lap-welded tubes that were calorized (coated with aluminum) by the General Electric Co. have been in operation for several weeks. The results to date of writing have been entirely satisfactory, especially as less carbon trouble has been experienced with these tubes than with like tubes not calorized.

Mr. A. J. Moxham, the president of the Aetna company, who has had extensive experience in the manufacture of steel, has given the problem of prolonging the life of the tubes his close personal attention. It is his opinion that the solution is to be found in the use of thick-walled cast tubes of high-carbon steels, the walls of which should be 1¼ to 1½ inches thick. The rate of heat transmission through walls of such thickness will be less than with the lap-welded tubes, which will tend to bring about a more uniform heat in the interior of the tubes. At the same time the thick walls should not present any difficulties in the way of maintaining satisfactory cracking temperatures. The great advantage expected to be gained results from the fact that cast steel of this character will not deteriorate under the influence of heat until the temperature becomes excessive. This means prolonged service and decreased cost of operation. The objection to the use of a cast-steel tube is the difficulty in making satisfactory castings. This is overcome in a large measure by having the walls of the thickness indicated. Several cast-steel tubes with walls three-fourths inch thick were tried out but did not give satisfactory results. The tubes were pitted after several days heating in the furnace. It is probable that cast-steel tubes of greater thickness would have given better satisfaction, as a better casting could be more easily obtained. Rolled-steel tubes have also been experimented with, and satisfactory results obtained up to the present writing. Because of the fact that rolled tubes at least 1 inch thick can be purchased in stock sizes at a less cost than that at which special cast-steel tubes can be had, it will probably be advisable to use rolled stock in new installations.

CONCLUSIONS REGARDING TUBES.

Based upon experience extending over a period of seven months, it is recommended that either rolled or cast steel tubes be used in lieu of lap-welded tubes. The walls of the tubes should be not less than 1 inch thick for maximum efficiency in operation.

GAS FORMATION.

The benzene-toluene process produces more than enough gas for continual operation of the cracking furnaces. Regardless of the character of oil used, 40 to 50 cubic feet of fixed gases is generated for each gallon of oil cracked. In order to maintain the proper temperature in the tubes a consumption of approximately 300 cubic feet of natural gas per hour is required for each tube or furnace. On the basis of a feed of 15 gallons per hour and 40 cubic feet of fixed gases per gallon of oil, a tube would generate 600 cubic feet of fixed gases per hour—twice the amount of gas required to heat the tube. And this takes no account of heat economies that will probably be developed at the plant in the future. These figures show that on any extensive scale of operation a large surplus of valuable illuminating and heating gas will be produced, which can be used for general plant purposes such as fuel under boilers and stills, or can be sold for industrial or domestic uses. This gas, owing to its high ethane and ethylene content, as previously pointed out, averages between 1,000 and 1,400 British thermal units per cubic foot of gas. Its heating value is equal to or greater than that of the best grades of natural gas, which furnish twice as many heat units per cubic foot as the same quantity of coal gas.

A plant treating 500 barrels of oil per day would thus produce 840,000 to 1,050,000 cubic feet of gas in 24 hours. At a rate of feed of 15 gallons per hour, and assuming that the oil is fed into the tubes 20 hours out of the 24, approximately 70 tubes would be required to convert this amount of material. The furnaces for the 70 tubes would consume not more than 500,000 cubic feet of gas, which would leave at least 300,000 to 500,000 cubic feet of gas available for other purposes. Stated in terms of calorific value a surplus of 360,000,000 to 600,000,000 British thermal units would be placed at the disposal of the plant operator for general use.

Very small amounts of gas are produced in the gasoline process as compared with the benzene-toluene process, consequently either natural gas, oil, or some other form of fuel would have to be employed to heat the cracking furnace.

The above figure of gas consumption per tube is based on operations as conducted to November, 1915. There is reason to believe that if a large combustion chamber with efficient preheating of air is provided, the amount of gas required per tube can be considerably decreased. It is believed that 250 cubic feet per hour can be regarded as a maximum figure.

COMPOSITION OF GAS.

The percentage of unsaturated hydrocarbons carried by this gas is of fundamental importance in considering availability for illuminating purposes. It has been shown from analysis of the gases, sampled in the course of the laboratory experiments, that a sufficient percentage of unsaturated hydrocarbons, or illuminants, is produced to make the fixed gas valuable for domestic use. During the development of the plant opportunity for extensive sampling of the gases produced was not afforded, as attention was necessarily concentrated on the production of toluene and benzene, and the character of the gas was regarded as being a problem to be taken up after operations had been placed on an efficient commercial basis. The results of analyses of four average samples of gases from the large cracking tubes when operated under different conditions of temperature and pressure are given in Table 65. The figures obtained show clearly that the gas is of the same value and character as that produced in the laboratory experiments, for which data have already been given.

TABLE 65.—*Analyses of five samples of gases from 10-tube furnaces.*

[Analyst, G. A. Burrell.]

Constituent.	Percentage contained in sample No.—				
	1	2	3	4	5
Unsaturated hydrocarbons	13.0	20.8	18.6	13.8	1.0
Benzene (C_6H_6)	.9	1.0	.8	1.3	1.2
Oxygen (O_2)	.5	.7	1.0	.5	.3
Carbon monoxide (CO)	.9	1.1	.7	.9	5.8
Carbon dioxide (CO_2)	.2	.3	.5	.2	.1
Methane (CH_4)	61.1	58.1	60.4	63.2	71.4
Ethane (C_2H_6)	8.9	7.3	6.3	8.6	3.5
Hydrogen (H_2)	11.8	8.7	11.6	9.4	15.9
Nitrogen (N_2)	2.7	2.0	.1	2.1	.8

It has been shown that if cracking is carried out under atmospheric pressure or diminished pressure (vacuum) a relatively greater amount of gas can be produced and a higher percentage of unsaturated hydrocarbons obtained. This fact suggests that there is a large field for the employment of the process, using diminished pressure, for enriching coke-oven gas from which the benzene and toluene have been removed to make it suitable for illuminating and heating purposes.

Oil gas produced by the process, using regular pressures, might also be used for carburetting water gas. Under conditions as in 1915 it will probably be cheaper for the operators of a coke-oven gas plant to extract all the crude benzene possible from the gases, rather than to attempt to leave a considerable percentage in the gas for the purpose of making it more marketable.

RECOVERY OF CRACKED OIL.

The percentage of recovered oil is dependent on the temperatures and pressures under which operations are conducted. The data obtained with the larger tubes are in no wise different from those obtained in the laboratory experiments except in such degree as is explainable by reason of inadequate condensation.

Although the average quantity of oil recovered in the benzene-toluene process ranged between 25 and 35 per cent, it will be found that with efficient condensation 35 per cent or more may be consistently obtained. In fact, with oils having a specific gravity of 0.90 and 0.91, such as those being recovered at the development plant in December, 1915, the percentage of recovery runs consistently between 40 and 50 per cent.

RESIDUUM OF RECOVERED OIL AFTER REMOVING LOW-BOILING FRACTIONS.

Up to the time of writing no opportunity has been afforded for the detailed study of that part of the recovered oil which distills above 175° C. It is known that this residuum carried up to 2½ per cent of naphthalene, on the basis of original oil, also undetermined percentages of diphenyl and anthracene. The proportion of the higher aromatic compounds could be increased, so far as percentage formation on the basis of original oil is concerned, by running the residuum through the tubes again or by operating at higher temperatures and pressures.

At times the general character of the products obtained from the tubes has varied widely. Several tubes at times have produced an oil of a bright emerald color, whereas oil of dark or reddish color was usually obtained. Just what conditions or constituents imparted this green color to the recovered product has not been determined.

The residuum contains a considerable percentage of valuable lubricating oils; in fact, in the early stages of operations the recovered oil distilling above 175° C. was used by the workmen for lubricating purposes and found to give excellent results. The percentage of refined lubricating oils obtainable and the general character and value of such oils remain to be determined.

It is hoped that the Bureau of Mines will be able to take up a systematic and extended study of the products formed in the process. From the experimental evidence there is no doubt but that many valuable and important facts concerning the nature of the recovered oil remain to be determined, and some of them may have an important industrial bearing.

COST DATA.

It is difficult to give cost data for a unit plant for either the gasoline or benzene-toluene process. Although several plants are being projected for 1916, only one plant has been constructed during the period for which data have been given. This installation is the one owned and operated by the Aetna company. The conditions under which this plant was erected were such as to make the costs unusually high. Consequently it would not be fair either to the company or to the process to quote such figures. Also their quotation is inadvisable because of the fact that it is recommended that the installation be changed in a number of particulars in any projected plant. These changes have been pointed out in connection with the discussion of various parts of the mechanical equipment.

The cost of a plant on a unit basis will depend largely on the size of the installation. Practically the same amount of piping, etc., will be required for a four-tube unit as would be needed for a plant twice the size. The cost per tube of the necessary plant accessories decreases with the number of tubes installed. A 200-barrel tank, for example, does not cost much more than a tank of half that capacity.

The erection of the furnaces constitutes only a part of the expense to be incurred. The tanks for the storage of raw and finished products, a still, agitators, and other equipment for a complete plant will constitute a much greater part of the expense. Costs of this character will be largely obviated if either or both processes are operated in connection with a petroleum refinery where such equipment is already available.

The cost of a gasoline plant will be considerably less than that of a benzene-toluene plant, as little or no carbon is formed and elaborate equipment for the removal and reception of this substance is not required; also a gas scrubber and a gas holder would not be needed, as the volume of fixed gases generated in making gasoline is small.

If the oil is preheated prior to being introduced into the tube, less heat will be required to bring the oil up to the desired cracking temperature. The amount of gas consumed will be less, and the size and cost of the furnace can be reduced accordingly. These are important considerations in estimating the amount of money needed for the erection of the plant. The furnaces necessarily will cost more if the oil is fed in cold.

According to estimates which have been made[a] the cost of a unit installation of four tubes for the benzene-toluene process, under conditions as in 1915, will average approximately $5,500 per tube. This sum includes the construction of a steel-frame building, foundations, structural work for furnaces, tubes, valves, oil meters, oil pumps for delivering oil to the tubes, motors for operating the stirring rods and fan for delivering preheated air to each furnace, gas burners, carbon pots, condenser, gas scrubber, oil and gas piping for furnaces, and miscellaneous equipment. The figure given does not include cost of storage tanks or equipment for the treatment of the furnace products. The cost of construction per unit of material treated decreases with increase in size of tubes. A furnace for an 18-inch tube will not cost any more than a furnace for a 10-inch tube, for example, but its production capacity will be more than double that of the 10-inch tube.

For the purpose of showing more clearly what the processes can accomplish the following tabular statements of yields are given. These figures are based upon the information and experience acquired during more than six months of experimentation and development. They are conservative if efficient means of condensing are to be employed and if the gases after leaving the condenser are to be washed or scrubbed. The figures are for a single-stage operation only, and do not take into consideration the total yields that might be obtained if the residuum, or a fraction thereof, above the upper limit of the benzene-toluene cut, were rerun through the tubes. The tables follow:

Total quantity of products recoverable by means of the benzene-toluene process per day, in a unit plant of 10 single-tube furnaces (tubes 8 inches in diameter by 11½ feet in length) at a rate of oil feed of 15 gallons per hour.

		Gallons.
Oil consumption per day (20 hours)		3,000
Recovered oil per day (35 to 50 per cent of original oil):		
Benzene recovered (6 to 8 per cent of original oil)	180	
Toluene recovered (6 to 8 per cent of original oil)	180	
Xylenes recovered (4 to 6 per cent of original oil)	120	
Gasoline recovered (6 to 8 per cent of original oil)	180	
Creosote oil and pitch,[b] (25 to 35 per cent of original oil)	340	
Total		1,050
Carbon produced per day (3 to 5 per cent of original oil)		[c]150
Gas produced per day (45 to 60 per cent of original oil)		[d]1,800
Total recovered oil, carbon, and gas		3,000

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[a] Furnished by courtesy of the Synthetic Hydro-Carbon Co. of Pittsburgh, Pa.

[b] Includes higher aromatics, such as cymene, diphenyl, naphthalene, anthracene, also valuable lubricating oil.

[c] 1,200 pounds.

[d] 120,000 cubic feet.

Total quantity of gasoline recoverable per day in a unit plant of 10 single-tube furnaces (tubes 8 inches in diameter by 11½ feet in length) at a rate of oil feed of 30 gallons per hour.[a]

		Gallons.
Rate of oil consumption per day (22 hours)		6,600
Gasoline recovered per day (25 to 30 per cent of original oil)	1,650	
Residuum (above 150° C.) recovered per day, available for rerunning (70 to 73 per cent of original oil)	4,620	
Loss per day (1½ to 3 per cent of original oil) in fixed gases and carbon)[b]	330	
Total		6,600

LARGE-SCALE GASOLINE PRODUCTION.

Opportunity has not been afforded for testing the gasoline process on a large commercial scale, as is the case of the benzene-toluene process. The Department of the Interior has, from time to time, announced its readiness to enter into an agreement for the large-scale use of the process similar to that which was made with respect to its companion process. Negotiations have been had at various times with different groups of oil refiners and different oil companies, but nothing in the way of concrete agreement satisfactory to both sides has resulted. The inability of the department to accede to the requests made for exclusive rights, or for recoupment of expenses incurred in the development of the gasoline process, has prevented reaching an understanding. At the time of writing licenses have been issued to four refining companies, each of which is about to install small experimental units.[c]

The Aetna Explosives Co. has been most courteous and obliging in extending facilities for conducting gasoline tests in the single-tube furnaces, during such times as the use of the furnaces could be spared for this purpose. Accordingly a considerable number of runs were made in the single tubes with the different oils available. Three oils were used in the experiments, one was a fuel oil from Pennsylvania-West Virginia crude petroleums and the other two were distillate oils, one having a boiling point ranging between 250° and 350° C., and the other between 200° and 300° C.

EXPERIMENTS IN 6-INCH BY 9-FOOT TUBE.

TESTS WITH DISTILLATE HAVING BOILING POINT OF 250° TO 300° C.

Table 66 gives the results of runs with the distillate boiling at 250° to 350° C. in a 6-inch by 9-foot tube at temperatures ranging between 550° and 650° C. and rates of feed varying from 13 to 26 gallons per hour. Both the percentage of recovered oil and the percentage of gasoline formed are abnormally low, which is due entirely to inefficient condensation. For this reason the figures published are to be regarded as suggestive rather than as definite evidence of the possibilities of the process. The greatly increased rate of feed in the gasoline process, as compared with the benzene-toluene process, caused a greater volume of gases to pass through the condenser in a given space of time and made the condensing apparatus available for these tests even more inadequate than it was in the case of the benzene-toluene process. With inefficient condensation the low-boiling hydrocarbons are readily lost. The amounts of recovered oil shown would have been 10 to 25 per cent greater if the condensing surface had been in proportion to the amount of gases generated. Even with this percentage of loss, the results show clearly the efficiency of the process as a means of producing gasoline from the higher boiling petroleum products.

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[a] Nature of reaction is so much simpler in gasoline process that less time is required and rate of oil feed accordingly can be doubled.

[b] This figure is high for large-scale operations.

[c] For form of license, see page 132.

Although the boiling points of the original oils ranged between 250° C. and 350° C., yet an average of 19.5 per cent of gasoline boiling below 150° C. was recovered in the runs reported. This percentage is on the basis of original oil and indicates that between 25 and 30 per cent could have been recovered if the condensing means had been adequate.

The runs were made at a uniform pressure of 250 pounds per square inch, but at varying rates of feed and temperatures. The results show that as the rate of feed is increased, with pressure and temperature kept constant, the percentage of gasoline on the basis of original oil decreases. At a temperature of 550° C. and a pressure of 250 pounds, and a rate of feed of 17 gallons per hour, 18.9 per cent of gasoline was recovered, whereas at 26 gallons per hour a yield of only 12.9 per cent was obtained. The same phenomenon holds true at the higher temperatures. For example, a rate of feed of 19 gallons per hour at 650° C. gives a total gasoline yield of 21.4 per cent, whereas an increase of 6 gallons per hour reduces the yield of gasoline by 4 per cent.

Another characteristic feature of these tests is the increased rate of feed which is possible with higher temperatures. A feed of 15 gallons per hour at a temperature of 550° C. gave the best results, whereas at 650° C. a rate of 19 gallons per hour gave an optimum recovery.

A temperature of 600 C. and a rate of feed of 17 gallons per hour is shown to have been the most favorable for this particular oil in this size of tube. The same oil in the large 8-inch tube gave optimum results at a temperature of 550° C. and a feed of 25 gallons per hour. Too much reliance should not be placed on the temperature indications, as conditions of operation did not permit accurate determination of furnace temperatures. It is doubtful whether the recorded difference in optimum conditions for the two tubes really existed. The results of the tests in the 6-inch by 9-foot tube are given in Table 66.

TABLE 66.—*Gasoline formed from cracking a distillate oil*[a] *in a single-tube furnace (tube 6 inches in diameter and 9 feet long) at a constant pressure of 250 pounds per square inch, with varying temperatures and rates of oil feed.*[b]

Number of runs.	Temperature, °C.	Rate of oil feed, gallons per hour.	Oil recovered.		Gasoline.		
			Per cent.	Specific gravity.	Percentage in recovered oil.	Specific gravity.	Percentage formed, on basis of original oil.
1.......	550	17	54	0.800	35.0	0.772	18.9
2.......	550	19	85	.800	20.0	.730	17.0
5.......	550	26	92	.800	14.0	.730	12.9
1.......	575	13	58	.815	32.0	.750	18.6
3.......	600	17	79	.830	36.0	.760	28.5
1.......	600	19	85	.810	26.7	.735	21.8
6.......	600	23	78	.810	25.0	.736	19.5
3.......	625	22	76	.815	27.0	.740	20.5
1.......	650	19	69	.820	31.0	.745	21.4
1.......	650	21	79	.816	24.0	.723	19.0
1.......	650	25	86	.828	20.0	.740	17.2

[a] Boiling point 250° C. to 350° C.

[b] Results low, due to insufficient condensation.

EXPERIMENTS WITH 8-INCH BY 10-FOOT TUBE.

The same oil was cracked in a number of experiments in an 8-inch by 10-foot tube. The data are assembled in Table 67. The average percentage of gasoline formed is only 18.8 as compared with 19.5 in the smaller tube. In the larger tube the average rate of feed was increased from 22 gallons to 30 gallons per hour. The greater volume of gases generated placed a heavier burden upon the already inadequate condenser, which contributed to the average lower yield obtained.

The low percentage of gasoline formed is, in the majority of yields reported in this table, clearly due to the excessive rate of feed, which did not allow sufficient opportunity for conversion. When the rate of feed was in proper relationship to the temperatures and pressure, it will be noted, that the yield of gasoline, despite the inefficient condensation, is considerably higher than that obtained in the laboratory experiments at Columbia University. The decreased yields, however, with the electrically heated furnace were due to the fact, as has already been mentioned, that a considerable percentage of the volume of oil used was necessarily consumed in building up the operating pressure. The quantity so consumed would, in a small charge, constitute a serious loss.

Temperatures above 575° C. are shown to be unfavorable as regards commercial production because, although a reasonably satisfactory percentage of conversion takes place, the percentage of recovered oil decreases, indicating that too large an amount of the original oil has gone to make fixed gases and carbon.

The decrease in gasoline formation at high rates of feed is clearly evidenced by a study of the results for a given temperature and pressure. The tests show that pressure is favorable for the formation of gasoline, an increase of 3 per cent in the yield being obtained at a temperature of 575° C. and a rate of 24 gallons per hour by changing the pressure from 200 to 300 pounds per square inch. The results in general bring out the fact previously mentioned that temperatures of from 500° to 575° C. are most favorable for gasoline production, all factors considered. Table 67 shows the results of the tests.

TABLE 67.—*Gasoline formed from cracking a distillate oil*[a] *in a single-tube furnace (tube 8 inches in diameter and 10 feet long), at varying temperatures, pressures, and rates of oil feed.*[b]

Number of runs.	Temperature, ° C.	Pressure, pounds per square inch.	Rate of oil feed, gallons per hour.	Oil recovered.		Gasoline.		
				Per cent.	Specific gravity.	Percentage in recovered oil, to 150° C. cut.	Specific gravity.	Percentage formed, on basis of original oil, up to 150° C. cut.
1	550	50	11	81	0.820	22.5	0.74	18.3
1	550	100	25	93	.808	16.8	.74	15.7
1	550	150	17	80	.825	29.25	.78	23.4
1	550	150	27	[100][c]	.810	18.0	.76	18.0
1	575	200	24	79	.820	22.8	.76	18.1
1	550	250	12	81	.805	26.8	.76	21.8
1	550	250	22	[98][c]	.800	13.8	.74	13.8
1	550	250	23	70	.860	38.5	.75	26.9
1	550	250	55	85	.835	19.8	.75	16.8
1	575	250	13	75	.820	20.5	.76	15.3
1	575	250	20	70	.848	32.5	.76	22.8
1	575	250	26	66	.825	38.0	.76	25.3
1	575	250	36	84	.814	23.3	.75	19.6
1	600	250	28	75	.815	29.25	.76	21.9
1	600	250	57	80	.839	16.5	.76	13.0
1	600	250	68	81	.841	16.3	.74	13.2
4	650	250	24	70	.830	28.0	.74	19.6
2	550	275	26	78	.815	29.0	.74	22.6
1	675	275	48	75	.815	20.8	.75	15.6
1	675	275	52	75	.815	19.0	.74	14.3
1	575	300	24	70	.820	31.3	.75	21.9
1	600	300	24	80	.815	22.0	.75	17.6

[a] Boiling point, 250° C. to 350° C.; specific gravity, 0.817 (41.3° B.)
[b] Results low, due to insufficient condensation.
[c] Obviously experimental error.

TESTS WITH DISTILLATE HAVING BOILING POINT OF 200° TO 300° C.

The results of a series of runs in a 6-inch by 9-foot tube with a different distillate oil are given in Table 68. The average yield of 21.5 per cent is more favorable than was obtained in either of the other series of experiments reported in Tables 66 and 67. The higher temperatures are again shown to be unfavorable for gasoline production. A satisfactory degree of conversion at such temperatures is offset by a considerable diminution in quantity of recovered oil, which is sufficient to condemn their use in a commercial plant.

TABLE 68.—*Gasoline formed from cracking a distillate oil in a single-tube furnace (tube 6 inches in diameter and 9 feet long) at varying pressures, temperatures, and rates of oil feed.*[a]

Number of runs.	Pressure, pounds per square inch.	Temperature, ° C.	Rate of oil feed, gallons per hour.	Percentage of oil recovered.	Gasoline.		
					Percentage in recovered oil.	Percentage formed, on basis of original oil.	Specific gravity.
1	125	600	18	79	38.0	30.0	0.730
1	125	650	12	....	36.0	....	.746
1	125	700	18	66	42.5	28.0	.735
4	250	550	25	91	11.2	10.2	....
1	250	550	33	91	15.0	13.7	.734
7	250	600	19	79	30.0	23.7	.748
3	250	600	24	80	26.4	21.1	.736
2	250	625	18	68	36.5	24.8	.752
2	250	625	24	73	27.7	20.2	.738
2	250	650	20	74	29.8	22.0	.731
1	250	650	25	71	31.0	22.0	.745

[a] Results low, owing to insufficient condensation.

DISTILLATION ANALYSIS OF ORIGINAL OIL.

[Specific gravity, 0.817; first drop at 170° C.]

Temperature of cut, ° C.	Distillation product.		Remarks.
	Proportion of original oil, by volume.	Specific gravity.	
	Per cent.		
200	2.0	....	Stuck at 290° to 296° C.
250	7.3	0.792	
300	57.0	.818	
350	28.0	.827	
Residue.	5.3	....	

TESTS WITH FUEL OIL.

The highest average conversion figures were obtained in experiments with a distillate fuel oil, data for which are presented in Table 69. The average yield of gasoline is nearly 3 per cent greater than in any of the other series of runs. It is to be noted, however, that the average percentage of recovered oil is lower. It is evident that in these runs the rate of feed was too low for the most efficient results. The results of the tests, which were made in an 8-inch by 10-foot tube, are given in Table 69.

TABLE 69.—*Gasoline formed from cracking a distillate fuel oil in a single-tube furnace (tube 8 inches in diameter and 10 feet long) at a constant pressure of 250 pounds per square inch, with varying temperatures and rates of oil feed.*[a]

Temperature, °*C.*	Rate of oil feed, gallons per hour.	Percentage of oil recovered.	Gasoline.		
			Percentage in recovered oil.	Percentage on basis of original oil.	Specific gravity.
575	12	75	30.0	22.5	....
575	15	79	25.1	19.8	....
600	12	71	31.3	22.2	0.76
625	12	77	40.0	30.8	.72
625	16	75	38.5	28.9	.75
650	12	74	36.3	26.8	.75
650	15	88	20.0	17.6	....
700	15	80	30.5	26.0	.78

[a] Results low owing to insufficient condensation.

DISTILLATION ANALYSIS OF ORIGINAL OIL USED.

[Specific gravity, 0.915 (23° B.); first drop at 300° C.]

Temperature of cut.	Distillation product.		
	Volume.	Proportion of original oil.	Specific gravity.
°*C.*	*C. c.*	*Per cent.*	
350	85	42	0.85
400	80	40	(*b*)

b Jelly-like.

CONCLUSIONS AS TO RESULTS OF TESTS.

In considering the figures assembled in the foregoing tables, it must be remembered that efficient condensation was lacking. The work was carried on during intervals when benzene-toluene experiments were being conducted with equipment erected to obtain indications as to benzene-toluene reactions, and not to determine maximum yields. Accordingly the condensing facilities were of the crudest type. The results can be considered only as suggestive of what can be accomplished under proper conditions. Viewed in this light they are of a distinctly favorable character, and justify optimistic belief in the large-scale commercial possibilities of the process.

GASOLINE PROCESS COMPARED WITH BENZENE-TOLUENE PROCESS.

In comparison with the benzene-toluene process, the gasoline process is simple in character. This may be more readily perceived by considering the order of hydrocarbon reactions as given below. The relative order is not only based on theory, but is in accordance with the evidence gained as the result of extended series of tests.

Order of Hydrocarbon Reactions.

Heavy petroleum hydrocarbons ⇄ Light petroleum hydrocarbons
(saturated and unsaturated).
⇅
Cymene, etc.
⇅
Xylene,
⇅
Toluene,
⇅
Benzene,
⇅
Naphthalene, Diphenyl, etc.
⇅
Anthracene, etc.
⇅ ⇅
Carbon. Gas.

The conversion from heavy hydrocarbons into lighter hydrocarbons involves only one step, and is easy of accomplishment, whereas the conversion into the lower boiling aromatic hydrocarbons requires that the heavier hydrocarbon molecules undergo a much more extensive series of changes before the desired products are obtained. This difference will indicate why the mechanical difficulties experienced in the first stages of development of the benzene-toluene process are nonexistent so far as the gasoline process is concerned. The lower temperatures necessary and the greatly decreased quantity of carbon produced eliminate serious trouble from overheating and clogging in gasoline manufacture.

Because of the greater ease with which gasoline can be made the installation used for benzene-toluene production will have double the capacity when used for conversion of heavy oil into gasoline. The duration of the gasoline reaction is less than half that required for the benzene-toluene reaction if the same size and length of tubes be used. It is manifest, therefore, that by introducing twice the volume of oil to be converted, the effect will be the same as decreasing the time factor by one-half.

The installation if used solely for gasoline production can be materially changed in many respects. The expensive carbon receptacles can be done away with, and the height of the furnaces above the ground materially reduced, thus lowering the cost of furnace construction. With the knowledge that has been gained of the nature of the reactions in the formation of gasoline and benzene and toluene there is no question as to the feasibility of both processes.

The total amount of gasoline that will be produced from a given quantity of oil depends on the number of times that the residuum above the gasoline fraction, after primary conversion, is rerun through the furnace. Each successive rerunning will give proportionately the same yields of gasoline as in the first operation. Aside from the amount of fixed gases produced, and the very small amount of carbon, the entire quantity of original oil can be converted into gasoline. In other words, on the basis of a barrel of 42 gallons, 33 to 37 gallons of gasoline can be recovered in from three to four runs.

In a commercial plant it would probably be found advisable to run the residuum above the gasoline fraction back into the storage tanks from which the original oil is fed into the tubes. This is a mere matter of detail, however, and there is no reason why the residuum, after being delivered to a suitable receiving tank, can not be fed direct into the tube without admixture with the original oil.

CONCLUSIONS AS TO GASOLINE PROCESS.

The large-scale experiments have fully confirmed the laboratory experiments and established the fact that the conversion into gasoline can be even more satisfactorily accomplished in a tube of greatly enlarged diameter and increased length than in the electrically heated 1½-inch tube. The conditions favorable for gasoline production are shown to be the same in the larger tubes as in the small tube, namely, a temperature of approximately 500° to 575° C. and a pressure of 250 to 300 pounds per square inch.

The gasoline process, therefore, can justly be considered as a success so far as conversion in the large tubes is concerned. The adaptation of the unit to refinery conditions is a matter of mechanical detail involving no inherent difficulties.

TERMINATION OF AGREEMENT BETWEEN GOVERNMENT AND AETNA COMPANY.

Early in September, 1915, the Department of the Interior was advised by the Aetna Explosives Co. that the plants were completed for the production of explosives from benzene and toluene obtained by the process of cracking in the vapor phase. In accordance with the express understanding between the company and the bureau, this necessitated the severance of the cooperative relations existing. This understanding was had to obviate any objection due to the utilization of Government employees in work involving the preparation of explosives. On the receipt of the above-mentioned notification, the Secretary of the Interior accordingly advised the Aetna company that the contract would be terminated. The Secretary's letter to this effect is given below:

AUGUST 24, 1915.

MY DEAR MR. MOXHAM: In accordance with your telephonic notice that you regard yourselves as now prepared to manufacture toluol and benzol upon a commercial basis by use of the Rittman process, I beg to advise you that from this time forward the agreement entered into between your company and myself is at an end. This, as you know, is in accordance with the understanding that we had that this plant was not to be used by us for any other than experimental purposes, and no employees of this department will further assist you in an effort to produce benzol and toluol.

Upon the issuance of the patent a license agreement will be made to your company, in accordance with our previous understanding. In the meantime, I am inclosing you a permit to use the process.

Very truly yours,

(Signed) FRANKLIN K. LANE.

Mr. A. J. MOXHAM,
President The Aetna Explosives Co. (Inc.),
2 Rector Street, New York City, N. Y.

The president of the Aetna Explosives Co. responded to this communication as follows:

AETNA EXPLOSIVES COMPANY, Inc.,
PRESIDENT'S OFFICE,
2 Rector Street, New York, September 10, 1915.

Hon. FRANKLIN K. LANE,
Secretary of the Interior, Washington, D. C.

MY DEAR MR. LANE: In view of your recent letter advising of the termination of the agreement between this company and the Department of the Interior, for the development of the Rittman benzol process, I thought this might be an opportune time for us to advise you as to our judgment concerning the present status of this process.

We believe that the progress thus far made in the development of the process has been such as to justify our placing the plant on a commercially operative basis. We have just completed arrangements to put this into effect.

It is true that there still remain various refinements which are to be worked out and installed in order to achieve the best possible results. Nevertheless, there seems to be no doubt in the minds of our staff at Pittsburgh as to the successful outcome of the process. It is our belief that within a few weeks it will be supplying benzol and toluol in quantities, and we feel very sure that greatly improved results will follow the refinements which are now being installed.

I take this occasion to assure you of our appreciation of the helpful attitude of the department, and of the assistance extended by it during the development period.

With kind regards, very truly, yours,

(Signed) A. J. MOXHAM,
President.

SUCCESS OF BENZENE-TOLUENE PROCESS.

In view of this communication, as well as the results of the large-scale operations, which have been given in detail, there can exist little doubt as to the success of the benzene-toluene process on a commercially operative scale. It has been proved that benzene and toluene can be produced in large quantities by this process. These products have been shown to be capable of being worked into the trinitrocompounds desired for making explosives. These nitro derivatives, or the derivatives thereof, are equally suitable for use in producing dyestuffs.

The claims which were made for the process at the outset have thus been fully verified by commercial results, and its future is dependent alone upon the perfection of the mechanical apparatus and the consequent reduction in labor and operating costs.

ACKNOWLEDGMENTS.

Particular thanks are due to Hon. Franklin K. Lane, Secretary of the Interior, for the great interest that he has at all times manifested in the development of the gasoline and benzene-toluene processes and for the hearty support that he has accorded the department employees who have been connected with the work.

Like measure of thanks is due to Mr. Van. H. Manning, Director of the Bureau of Mines, for his sympathetic support and hearty cooperation. Without his confidence in the future of the processes, they would still be in the laboratory stage of development and their possibilities in large-scale operations would still remain a matter of conjecture and theory.

President Nicholas Murray Butler, of Columbia University, by his kindness in extending the full use of the facilities of the university for the experiments, and by extending all possible privileges and courtesies, greatly facilitated the work.

Credit is also due in a large measure to Mr. W. A. Williams, chief of the petroleum division of the Bureau of Mines, for advice and assistance extended. He has kept in personal touch with the development work and has given freely of his time and knowledge in order to make it a success.

Acknowledgments are due to Prof. Milton C. Whitaker, of Columbia University, for practical advice and personal cooperation in the progress and development of the experimental work leading to the discovery of the processes; to Dr. George A. Hulett, of Princeton University, consulting chemist of the Bureau of Mines, for hearty cooperation and support throughout the long course of experimentation; and to Profs. J. R. L. Morgan, F. J. Metzgar, M. T. Bogart, and Hal T. Beans, of Columbia University, for practical suggestions and advice, and valuable time given to informal discussions of the principles involved in the problem. Thanks are also due to Prof. Gellert Alleman, of Swarthmore College, for constructive advice and assistance and material aid in the successful working out of the problem.

The authors desire to make particular acknowledgment of their indebtedness to Mr. A. J. Moxham, president of the Aetna Explosives

Co., for his great interest in the development of the benzene-toluene process and for the time and personal attention that he has given to the related problems. The unfailing courtesy and great consideration that he has uniformly extended, in spite of the numerous discouragements and serious difficulties incident to the early stages of the development operations, have made the cooperative work a genuine pleasure. Acknowledgments are also due to Mr. Egbert Moxham, general manager, and to Maj. J. T. Crabbs, division manager, of the Aetna company, for valuable assistance extended in the analysis and solution of operating difficulties and for facilities given in the accumulation of data concerning plant operations.

Especial acknowledgment is also tendered to Mr. Pennock Hart, president, and Mr. E. H. Haslem, general manager, of Mackintosh, Hemphill & Co., of Pittsburgh, Pa., for the aggressive manner in which the perplexing problems incident to the development of an industrial process from the laboratory to a commercially operative scale have been taken up and successfully solved. Too much credit can not be given to them and to Messrs. C. C. Stutz and S. McMillan and other members of their organization for the great personal interest taken and earnest efforts expended.

The authors are peculiarly indebted to Dr. F. L. Slocum for his valuable assistance and advice during the early stages of development work; also to Mr. Darwin S. Wolcott, for aid and advice and for the faith that he has at all times manifested in the success of the processes. The earnest personal efforts of Dr. David T. Day, consulting chemist of the Bureau of Mines, in the difficult initial work at the outset of the factory development are appreciatively acknowledged.

Thanks are also due to members of the working force of the Aetna Chemical Co. plant, both those connected with the mechanical operation of the process and those connected with the laboratory, for the whole-souled enthusiasm with which they have thrown themselves into the work and contributed unsparingly of time and energy for the successful development of the process.

Particular thanks are due to Dr. Gustav Egloff, who is in charge of the Aetna company's laboratory at the development plant, for valuable services in connection with the industrial development of the benzene-toluene process and for aid in the preparation of this paper.

TABLES OF REFERENCE.

For convenience of reference a number of tables are given which contain figures of use to oil men. The Fahrenheit-centigrade conversion table (No. 71) is for approximate transformations. Table 70, for converting Baumé degrees to specific gravities, is taken from Van Nostrand's Chemical Annual. It makes use of the Baumé scale, approved by the United States Bureau of Standards, which is in general, though not universal, use among oil men in this country. The use of Baumé degrees as a measure of specific gravity is to be deplored, as the scale is not based on any simple principle, and in addition is not definitely fixed. At least one set of tables, largely used by oil men, employs a scale different from that officially accepted. The use of Baumé readings is, however, so firmly established that there is little hope that the scale will be replaced by the more logical system of specific gravities which represent ratios based on the density of water.

Tables 72 to 83 include data concerning the properties of various hydrocarbons which may be of importance to those who work with petroleum. All of these tables are taken directly from Redwood's[a] treatise, except Table 72, which is from an article by Rittman and Egloff[b] containing figures collected from various sources.

TABLE 70.—*Conversion table—Baumé degrees (American standard) to specific gravities (at 60° F.).*

[Specific gravity $= \frac{140}{130-B^{\circ}}$ for liquids lighter than water.]

Degrees Baumé.	Specific gravity.	Degrees Baumé.	Specific gravity.	Degrees Baumé.	Specific gravity.	Degrees Baumé.	Specific gravity.
10	1.000	31	0.870	51	0.773	71	0.696
11	.993	32	.864	52	.769	72	.693
12	.986	33	.859	53	.765	73	.690
13	.979	34	.854	54	.761	74	.686
14	.972	35	.848	55	.757	75	.683
15	.965	36	.843	56	.753	76	.680
16	.959	37	.838	57	.749	77	.676
17	.952	38	.833	58	.745	78	.673
18	.946	39	.828	59	.741	79	.670
19	.940	40	.823	60	.737	80	.667
20	.933	41	.819	61	.733	81	.664
21	.927	42	.814	62	.729	82	.660
22	.921	43	.809	63	.725	83	.657
23	.915	44	.805	64	.722	84	.654
24	.909	45	.800	65	.718	85	.651
25	.903	46	.795	66	.714	86	.648
26	.897	47	.791	67	.711	87	.645
27	.892	48	.786	68	.707	88	.642
28	.886	49	.782	69	.703	89	.639
29	.880	50	.778	70	.700	90	.636
30	.875						

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[a] Redwood, Boverton, Petroleum, London, vol. 1, 1913, pp. 243–245, 248–250, 255–256; vol. 2, p. 202.

[b] Rittman, W. F., and Egloff, Gustav, Physical constants of gas oils and derived tars: Jour. Ind. and Eng. Chem., vol. 7, June, 1915, pp. 481–484.

TABLE 71.—*Conversion table, centigrade to Fahrenheit degrees.*

Centigrade degrees.	Fahrenheit degrees.	Centigrade degrees.	Fahrenheit degrees.
−20	−4	225	437
−10	14	250	482
0	32	275	527
10	50	300	572
20	68	350	662
30	86	400	752
40	104	450	842
50	122	500	932
60	140	550	1,022
70	158	600	1,112
80	176	650	1,202
90	194	700	1,292
100	212	750	1,382
125	257	800	1,472
150	302	850	1,562
175	347	900	1,652
200	392		

TABLE 72.—*Physical properties of various oil and tar constituents.*

Constituent.	Boiling point.	Specific gravity.	Refractive index at — °C.	
			Temp.	Value.
Paraffins:	*°C.*			
Hexane	68	0.665	14.8	1.378
Heptane	92 to 94	.709	15.0	1.404
Octane	125.5	.707	15.1	1.401
Nonane	136 to 138	.740	15.0	1.421
Decane	173	.728	14.9	1.411
Tetradecane	[a] 126	.770	20.0	1.431
Hexadecane	[a] 151	.780	20	1.436
Octodecane	[a] 174	.779	27	1.439
Olefins:				
Hexylene	....	.689	15.2	1.400
Octylene	....	.726	16	1.416
Decylene	....	.773	17	1.439
Aromatic hydrocarbons:				
Benzene	80.4	.876	20	1.501
Toluene	110.3	.871	14.7	1.499
Ethyl benzene	134.0	.875	14.5	1.499
o-xylene	142.0	.885	14.1	1.508
m-xylene	139.0	.869	15.7	1.500
p-xylene	138.0	.866	14.7	1.499
N-propyl benzene	158.5	.866	15.7	1.494
Isopropyl benzene	153.0	.863	15.1	1.495
Mesitylene	164.5	.865	14.6	1.497
Pseudocumene	170.0	.883	....	1.507
Cymene	175.0	.862	13.7	1.511
Isobutyl benzene	167.0	.872	17.5	1.496
Naphthenes:				
Cyclohexane	80.8	.779	19.5	1.427
Methylpentamethylene	72.0	.750	20.0	1.411
Methylhexamethylene	101.0	.769	20.0	1.424
Isohexamethylene	101.0	.756	19.0	1.418
Dimethylpentamethylene	93 to 96	.754	20.0	1.413
Hexahydro-m-xylene	117 to 118	.758	17.0	1.419
Ethyl cyclohexane	130	.799	4.0	1.433
1, 3 dimethylhexamethylene	120	.769	20.0	1.423
1, 1 dimethylhexamethylene	....	.773	18.0	1.424
1, 4 dimethylhexamethylene	118.2	.769	20.0	1.424
3-methyl-1, 2, 5-hexamethylene	142 to 144	.781	20.0	....
1, 3, 5-trimethylcyclohexane	134 to 136	.788	4.0	1.426
Mononaphthene	170 to 172	.773	16.0	1.433
1, 3 diethylhexamethylene	....	.796	22.0	1.439
Isobutylcyclohexane	157	.804	21.0	1.447

[a] At 12 mm. pressure.

TABLE 73.—*Properties of paraffins from Pennsylvania petroleum.*

First series.				Second series.			
Symbol.	Boiling point.	Specific gravity at 0° C.	Vapor density.	Symbol.	Boiling point.	Specific gravity at 0° C.	Vapor density.
	°*C.*				°*C.*		
C_4H_{10}	....	0.600	2.110	C_4H_{10}	8 to 9	0.611	....
C_5H_{12}	30.2	.640	2.538	C_5H_{12}	37.0	.645	2.514
C_6H_{14}	61.3	.676	3.053	C_6H_{14}	68.5	.689	3.038
C_7H_{16}	90.4	.718	3.547	C_7H_{16}	98.1	.730	3.551
C_8H_{18}	119.5	.737	3.992	C_8H_{18}	127.6	.752	3.990
C_9H_{20}	150.8	.756	4.600				

TABLE 74.—*Properties of hydrocarbons in Pennsylvania petroleum.*

Hydrocarbon.	Symbol.	Boiling point.	Melting point.
		°*C.*	°*C.*
Tridecane	$C_{13}H_{28}$	226	....
Tetradecane	$C_{14}H_{30}$	236 to 238	....
Pentadecane	$C_{15}H_{32}$	256 to 257	....
Hexadecane	$C_{16}H_{34}$	274 to 275	....
Heptadecane	$C_{17}H_{36}$	288 to 289	10
Octodecane	$C_{18}H_{38}$	300 to 301	20
Nonodecane	$C_{19}H_{40}$	210 to 212 (50 mm.)	33 to 34
Heneicosane	$C_{21}H_{44}$	230 to 231 (50 mm.)	40 to 41
Hydrocarbon, liquid at 10° C	$C_{22}H_{44}$	240 to 242 (50 mm.)	....
Docosane	$C_{22}H_{46}$	....	44
Hydrocarbon, liquid at 10° C	$C_{23}H_{46}$	258 to 260 (50 mm.)	....
Tricosane	$C_{23}H_{48}$	....	45
Hydrocarbon, liquid at 10° C	$C_{24}H_{48}$	272 to 274 (50 mm.)	....
Tetracosane	$C_{24}H_{50}$	....	48
Hydrocarbon, liquid at 10° C	$C_{26}H_{52}$	280 to 282 (50 mm.)	....
Pentacosane	$C_{25}H_{52}$	....	53 to 54
Hydrocarbon, liquid at 10° C	$C_{27}H_{52}$	292 to 294 (50 mm.)	....
Hexacosane	$C_{26}H_{54}$	....	58
Hydrocarbon, liquid at 10° C	$C_{28}H_{54}$	310 to 312 (50 mm.)	....
Octocosane	$C_{28}H_{58}$	....	60

TABLE 75.—*Properties of hydrocarbons in Ohio (Trenton limestone) petroleum.*

Series.	Symbol.	Boiling point.	Pressure at which boiling point was recorded.	Specific gravity at 20° C.
		°*C.*	*Mm.*	
C_nH_{2n}	$C_{12}H_{24}$	211–213	760	0.7970
C_nH_{2n}	$C_{13}H_{26}$	223–225	760	.8055
C_nH_{2n}	$C_{14}H_{28}$	138–140	30	.8129
C_nH_{2n}	$C_{15}H_{30}$	152–154	30	.8204
C_nH_{2n}	$C_{16}H_{32}$	164–168	30	.8254
C_nH_{2n}	$C_{17}H_{34}$	177–179	30	.8335
C_nH_{2n-2}	$C_{19}H_{36}$	198–202	30	.8364
C_nH_{2n-2}	$C_{21}H_{40}$	213–217	30	.8417
C_nH_{2n-2}	$C_{22}H_{42}$	224–227	30	.8614
C_nH_{2n-2}	$C_{24}H_{46}$	237–240	30	.8639
C_nH_{2n-4}	$C_{23}H_{42}$	253–255	30	.8842
C_nH_{2n-4}	$C_{24}H_{44}$	263–265	30	.8864
C_nH_{2n-4}	$C_{25}H_{46}$	275–278	30	.8912

TABLE 76.—*Properties of hydrocarbons in Canadian petroleum.*

Compound.	Symbol.	Boiling point.	Pressure at which boiling point was recorded.	Specific gravity at 20° C.
		°*C.*	*Mm.*	
Hydrocarbon	$C_{12}H_{24}$	216		
Chloride	$C_{12}H_{23}Cl$	160	15	0.9145
Hydrocarbon	$C_{13}H_{26}$	228-230		.8067
Chloride	$C_{13}H_{25}Cl$	165		.9221
Hydrocarbon	$C_{14}H_{28}$	141-143	50	.8096
Chloride	$C_{14}H_{27}Cl$	180	15	.9288
Hydrocarbon	$C_{15}H_{30}$	159-169	50	.8192
Chloride	$C_{15}H_{29}Cl$	190	15	.9358

TABLE 77.—*Properties of hydrocarbons in California petroleum.*

Symbol.	Boiling point at 60 mm. pressure.	Specific gravity at 20° C.
	°*C.*	
$C_{13}H_{24}$	150-155	0.8621
$C_{16}H_{30}$	175-180	.8808
$C_{17}H_{30}$	190-195	.8919
$C_{18}H_{32}$	210-215	.8996
$C_{24}H_{44}$	250-255	.9299
$C_{27}H_{46}$	310-315	.9451
$C_{29}H_{50}$	340-345	.9778

TABLE 78.—*Properties of naphthenes from petroleum.*

Symbol.	Source and authority.	Boiling point.	Specific gravity at °C.
		°*C.*	
C_6H_{12}	Hexahydrobenzene (Kijner)	69	0.7539
	Russian petroleum		
C_7H_{14}	Hexahydrotoluene (Lossen)	97	.772
	Russian petroleum		
	From the products of dry distillation of colophony (Renard)	95 to 98	.742 $\frac{20^\circ}{20^\circ}$
C_8H_{16}	Hexahydro-m-xylene (Wreden)	115 to 120	.777
	Russian petroleum (Wreden)	122 to 124	.7835
	From colophony	120 to 123	.764 $\frac{19^\circ}{19^\circ}$
C_9H_{18}	Hexahydromesitylene (Bayer)	135 to 138	
	Hexahydrocumene (Konowaloff)	135 to 138	.7812
	From petroleum	135 to 136	.7808
	Hexahydropropylbenzene (Tchitchibabin)	140 to 142	.7811
$C_{10}H_{20}$	Dodecahydronaphthalene (Wreden)	153 to 158	.802
	From petroleum	160 to 162	.7808 $\frac{17.40^\circ}{40^\circ}$
	.....do	168 to 170	.8073 to 0.814 $\frac{15^\circ}{15^\circ}$
	From menethene	168.5 to 170	.797 $\frac{15^\circ}{15^\circ}$
	From terpene hydrate	168.5 to 170	.797 $\frac{15^\circ}{15^\circ}$
	From camphor (Starodubsky)	167 to 169	.8114 $\frac{0^\circ}{0^\circ}$
	Tetrahydroterpene (Orloff)	162 to 167	.806 $\frac{0^\circ}{0^\circ}$
$C_{11}H_{22}$	From petroleum	179 to 181	.8019 $\frac{16.2^\circ}{40^\circ}$
$C_{12}H_{24}$	.....do	197	.8120 $\frac{18.4^\circ}{40^\circ}$
$C_{14}H_{28}$	.....do	240 to 241	.8215 $\frac{18.6^\circ}{40^\circ}$
$C_{15}H_{30}$	.....do	246 to 248	.8210 $\frac{18.8^\circ}{40^\circ}$

TABLE 79.—*Properties of $C_nH_{2n}+2$ hydrocarbons.*

Symbol.	Specific gravity.	Refractive index.	Boiling point.	Pressure.	Melting point.	Authority.
			° *C.*	m. m.	° *C.*	
C_4H_{12} (iso)			0	760		Mabery.
C_5H_{12} (normal)	0.6250—25°/25° [a]		36.3	760		Young.
	.6261—0°/4°					
	.6454—0°/4°					
C_5H_{12} (iso)	.6392—0°/4°		27.95	711		Young.
C_6H_{14} (normal)	.6771—0°/4°		68.95	711		Do.
C_6H_{14} (iso)	.6730—0°/4°		61.00	711		Do.
C_7H_{16} (normal			98.40	711		Do.
C_7H_{16} (iso)	.6969—0°/4°		90.30	711		Do.
C_8H_{18} (normal)	.7188—20°/20°		125.00	760		Do.
C_8H_{18} (iso)	.7190—20°/20°		119.50	760		Mabery.
C_9H_{20} (normal)			151.00	760		Do.
$C_{10}H_{22}$ (normal)	.7479—20°/20°		163-164	760		Do.
$C_{10}H_{22}$ (iso)	.7467—20°/20°		173-174	760		Do.
$C_{11}H_{24}$	.7581—20°/20°		196-197	760		Do.
$C_{12}H_{26}$ (normal)	.7676—20°/20°		214-216	760		Do.
$C_{13}H_{28}$	.7834—20°/20°	1.451	226	760		Do.
$C_{14}H_{30}$	.7814—20°/20°	1.436	236-238	760		Do.
$C_{15}H_{32}$	.7896—20°/20°	1.4413	256-257	760		Do.
$C_{16}H_{34}$	.7911—20°/20°	1.4413	274-275	760		Do.
$C_{17}H_{36}$	.8000—20°/20°	1.4435	288-289	760	10	Do.
$C_{18}H_{38}$	.8017—20°/20°	1.440	300-301	760	20	Do.
$C_{19}H_{40}$	.8122—20°/20°	1.4522	210-212	50	33-34	Do.
$C_{21}H_{44}$			230-231	50	40-41	Do.
$C_{22}H_{46}$	.7796—15°		240-242	50	44	Do.
$C_{23}H_{48}$	.7900—60°		258-261	50	45	Do.
$C_{24}H_{50}$	.7902—60°		272-274	50	48	Do.
$C_{25}H_{52}$	.7941—60°		280-282	50	53-54	Do.
$C_{26}H_{54}$	.7977—60°		292-294	50	58	Do.
$C_{28}H_{58}$	.7945—70°		310-312	50	60	Do.
$C_{31}H_{64}$	.7992—70°		328-330	50	66	Do.
$C_{32}H_{66}$	.8005—75°		342-345	50	68	Do.
$C_{34}H_{70}$	.8009—80°		366-368	50	72	Do.
$C_{35}H_{72}$	.80052—80°		380-384	50	76	Do.

[a] Specific gravity at 25° C., compared with that of water at 25° C.

TABLE 80.—*Properties of certain monocyclic polymethylenes and hydrocarbons.*

MONOCYCLIC POLYMETHYLENES C_nH_{2n}.

Symbol.	Compound.	Specific gravity.	Refractive index.	Boiling point.	Pressure.	Authority.
				° *C.*	*Mm.*	
C_5H_{10}	Pentamethylene	0.7000 at 0°/4°		50	760	Young.
C_6H_{12}	Methylpentamethylene	0.7660 at 0°/4°		72	760	Do.
C_6H_{12}	Hexamethylene	0.7722 at 0°/4°		80.6	760	Do.
C_7H_{14}	Dimethylpentamethylene	0.7543 at 20°/4°		94	760	Do.
C_7H_{14}	Methylhexamethylene	0.7964 at 20°/4°		102	760	Do.

HYDROCARBONS C_nH_{2n}.

Symbol.	Compound.	Specific gravity.	Refractive index.	Boiling point.	Pressure.	Authority.
$C_{21}H_{42}$		0.8424 at 20°/20°				
$C_{22}H_{44}$		0.8262 at 20°/20°	1.454	240 to 242	50	Mabery.
$C_{23}H_{46}$		0.8569 at 20°/20°	1.4714	258 to 260	50	Do.
$C_{24}H_{48}$		0.8598 at 20°/20°	1.4726	272 to 274	50	Do.
$C_{26}H_{52}$		0.8580 at 20°/20°	1.4725	280 to 282	50	Do.

HYDROCARBONS C_nH_{2n-2}.

Symbol.	Compound.	Specific gravity.	Refractive index.	Boiling point.	Pressure.	Authority.
$C_{27}H_{52}$		0.8688 at 20°/20°	1.4722	290 to 294	50	Mabery.
$C_{28}H_{54}$		0.8694 at 20°/20°	1.4800	310 to 312	50	Do.

TABLE 81.—*Solid hydrocarbons in Pennsylvania petroleum and their physical properties.*

Hydrocarbon.	Symbol.	Melting point.	Specific gravity.
		° C.	
Tetracosane	$C_{24}H_{50}$	50 to 51	0.7900 at 60° C.
Hentricontane	$C_{31}H_{64}$	66	0.7997 at 70° C.
Dotricontane	$C_{32}H_{66}$	67 to 68	0.8005 at 75° C.
Tetratricontane	$C_{34}H_{70}$	71 to 72	0.8009 at 80° C.
Pentatricontane	$C_{35}H_{72}$	76	0.8052 at 80° C.

TABLE 82.—*Hydrocarbons in commercial paraffins, and their physical properties.*

Hydrocarbon.	Symbol.	Melting point.	Specific gravity at 60° C.
		° C.	
Tricosane	$C_{23}H_{48}$	48	0.7886
Tetracosane	$C_{24}H_{50}$	50–51	
Pentacosane	$C_{25}H_{52}$	53–54	.7941
Hexacosane	$C_{26}H_{54}$	55–56	.7968
Octocosane	$C_{28}H_{58}$	60	
Nonocosane	$C_{29}H_{60}$	62–63	

TABLE 83.—*Relation of specific gravity to melting point of paraffins.*

Sample No.	Origin of sample.	Specific gravity of paraffin.		Solidifying point of paraffin.
		Solid at 15.5° C.	Liquid at 99° C.	
				° C.
1	Shale oil	0.8666	0.7481	44.0
2	.....do	.8961	.7494	47.0
3	.....do	.9000	.7517	52.0
4	.....do	.9111	.7572	58.5
5	American petroleum	.9083	.7535	53.8
6	Ozokerite		.7531	61.5
7	Rangoon tar	.8831	.7571	40.0

CHARACTER OF BIBLIOGRAPHY.

A general review of the literature relating to the manufacture of gasoline and benzene and toluene by the cracking of heavy hydrocarbons has not been attempted by the authors, as such a review, however valuable in and of itself, would have consumed too much time and space.

In lieu of such a review, Mr. M. S. Howard, librarian in charge of the Pittsburgh library, Bureau of Mines, has compiled and arranged a comprehensive bibliography of the subjects relevant to the discussion in this paper. The bibliography deals with the literature pertaining to the recovery of benzene, toluene, and related products from coal distillation; with that relating to methods of analyzing and purifying these substances; and with that pertaining to the production of gasoline and aromatic hydrocarbons from petroleum.

It has been deemed advisable to repeat references to articles of general character that treat of more than one subject.

The fact that, as far as is known, no previous attempt has been made to compile a complete and comprehensive list of references dealing with all phases of the production of benzene and toluene and the decomposition of heavy hydrocarbons into low-boiling gasoline hydrocarbons, should make the bibliography of value to workers in these fields.

The bibliography contains the important, easily accessible references from 1890 to September, 1915, on the recovery of benzene, toluene, and xylene from coal distillation, and nearly all references of value before 1890. The section on the production of benzene and toluene, and gasoline, from petroleum contains all references of value that came to the authors' notice to September, 1915.

References to analytical methods for determining pure hydrocarbons are in general not included, except those containing material on the detection or elimination of impurities in the crude products.

Attention is called to the words benzol, benzene, toluol, and toluene, which are spelled as in the article or paper cited.

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RECOVERY OF BENZENE, TOLUENE, AND RELATED PRODUCTS FROM COAL DISTILLATION.

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2. Bagley, D. C. The recovery of benzol from coke-oven gases. Chem. Eng., vol. 14, 1911, pp. 448–451; Iron Age, vol. 88, Oct. 5, 1911, pp. 756–759; Iron and Coal Trades Rev., vol. 83, 1911, pp. 235–237. Describes a Still plant for benzol recovery, the system being that for 90 per cent of benzol recovery in Germany. Oil for absorption is sprayed into the scrubbers, through which the ammonia-freed and cooled gases pass. The oil and benzol are separated by heating and by the action of live steam. Crude benzol, collected in a storage tank, is pumped to a primary still. Desired fractions are obtained by distillation. The uses of various products, as benzol, toluol, and xylol, are also briefly described. The yield of pure benzol obtained from crude benzol varies from 60 to 75 per cent.
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benzol in coal gas may be recovered without unduly impoverishing the gas. On this basis the United Kingdom could produce from gas works 12,000,000 gallons of benzene per annum, and from coke ovens 60,000,000 gallons.

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26. GWIGGNER, A., and SCHULZ, R. Ueber Nebenproduktengewinnung aus Generatorgas: Stahl und Eisen, Jahrg. 33, Bd. 2, Oct. 2, 1913, pp. 1652–1654. Discussion of the paper by Schulz in Stahl und Eisen, Jahrg. 33, Bd. 2, July 24, 1913, pp. 1221-1225.

27. HAARMANN, —. Über die Bedeutung und die Aussichten der Nebenprodukten-Industrie der Steinkohle. Glückauf, Jahrg. 42, Bd. 1, Apr. 7, 14, 1906, pp. 418–426, 445–455. Discusses production and uses of benzol. In Westphalian practice 4 kilograms of benzol and 0.9 kilograms of toluol per ton of coal coked are obtained. Statistics of production and prices in Germany from 1896 to 1904 are given. In 1904 Germany could have produced 70,000 tons of benzol.

28. HEINZERLING, C. Benzol winning from coke-oven gases. Mineral Industry, vol. 5, 1896, pp. 199–201; Jour. Soc. Chem. Ind., vol. 16, May 31, 1897, p. 432; Jour. Gasbel, Jahrg. 40, May 22, 1897, pp. 333–336. Method taken from Chemische Rundschau, 1897. Heinzerling has patented a method of separating benzol from coke-oven gases by condensation at −30° to −40° C. and expansion, by which the gas is cooled to −70° or −80° C., the benzol being deposited in tubes, which are duplicated, so that one set is always available. At −70° C. the vapor tension of benzol is zero. A diagram of the apparatus showing compression and expansion cylinders is given. The subject of costs and power required is discussed.

29. IRWIN, WILFRID. Determination of the vapor tensions of the condensable hydrocarbons in coal gas; its relation to the question of naphthalene deposits and enrichment with benzene. Jour. Soc. Chem. Ind., vol. 18, Feb. 28, 1899, pp. 109, 110. Gives condensable products from gas, with boiling points and yields. High temperature carbonization favors the production of aromatic hydrocarbons, but produces more naphthalene. Discusses vapor tension of hydrocarbons, and the enrichment of gas.

30. JONES, L. C. Coal and its by-products. Jour. Franklin Inst., vol. 177, May, 1914, pp. 511–531. Discusses origin, composition, and coking quality of coal. A ton of coal produces 1½ to 2½ gallons of mixed light oils. The influence of temperature upon the yields and character of the by-products from average coking coal is shown by a chart. Experiments indicate a maximum yield of light oil at about 500° C. to 550° C.

31. JÜNGST, ERNST. Nebenproduktengewinnung bei der Kokserzeugung. Glückauf, Jahrg., 50, March 28, 1914, pp. 495–498; Gas World, vol. 60, May 2, 1914, Coke section, p. 22. Summary of production of tar, benzol, and ammonia compounds in Germany from 1908 to 1912. Gives imports and exports of by-products.

32. KRÄMER, G. Ueber Benzol, und seine Bedeutung als Leuchtstoff. Jour. Gasbel., Jahrg. 40, June 5, 12, 1897, pp. 369–372; 385–388; Jour. Gas Lighting, vol. 69, June 22, 1897, pp. 1421–1423. On distilling coal, 8 per cent of the benzol remains in the tar, and 92 per cent goes into the gas. Good coal tar yields 1½ per cent of benzol and toluol together, but only about 1 per cent is obtained in practice. Benzol may be obtained as a by-product in the compression of heavy oil-gas, but can not be produced alone. Article also describes recovering of benzol from coke-oven gas, and discusses use for illuminating purposes.

33. LUNGE, GEORG. Coal tar and ammonia; 1909. Chapter entitled, "Light Oil and Crude Naphtha" (pp. 665–706), describes the methods of working up light oil and gives a digest of the more important work on this subject. Chapter entitled, "Working Up Light Naphtha Into Final Products" (pp. 707–809), describes the manufacture of commercial benzol products, and the preparation of pure benzene, toluene, and xylene. The latter part of the chapter is devoted to testing benzol, to estimating carbon bisulphide, thiophene, sulphur, xylenes, and other hydrocarbons, and to applications of benzol and naphtha. Chapter entitled, "Recovery of Benzol from Coal Gas" (pp. 36–57), Discusses various methods of benzol recovery, and details of a benzol washing plant according to Hirzel's system. Three additional sections are: Recovering the benzol from coal gas as nitrobenzene, composition of benzol obtained from coal gas, estimation of benzene and its congeners in coal gas. The many references in these chapters form an excellent bibliography of the earlier work on benzol.

34. MEISSNER, C. A. The modern by-product coke oven. Senate Document 145. 63d Cong., 1st sess., 1913. Recovery of by-products in the modern by-product coke-oven plant. Met. and Chem. Eng., vol. 11, August, 1913, pp. 454–458. Contains diagram of an apparatus for the recovery of benzol and a short paragraph on methods of recovery. Light oil used for scrubbing has specific gravity of about 0.8. Benzol is distilled from the scrubbed mixture, steam being the heating medium. The benzol obtained is about 50 per cent benzol. Usually the higher the volatile matter in the coal, the greater the yield of benzol.

35. MÜLLER, E. Absorption von Benzol in Wasser und Paraffinöl. Jour. Gasbel., Jahrg. 41, 1898, pp. 433–435. Tables show the amount of benzol absorbed from air carrying 1 to 8 per cent benzol, at temperatures of 10° to 18°. The time

was 2 to 8 hours, and the rapidity of passage 1.43 to 2.50 cubic centimeters per second. It was demonstrated that cooling aids the absorption of benzol by paraffin oil.

36. Muspratt, —. Theoretische praktische Chemie. Aufl. 4, 1905, Bd. 8, pp. 17–50. Contains a chapter on light oil and its products that treats of manufacture, uses, and tests of benzol, toluol, and xylol. The distillation method of Kraemer and Spilker is described, and use of the Le Bel Henninger fractionating column. Boiling-point curves for mixtures of benzene hydrocarbons are included.

37. Norton, T. H. Dyestuffs for American textile and other industries. Bureau of Foreign and Domestic Commerce. Special Agents Series No. 96. 1915. 57 pp. A long report on the dyestuff situation in the United States in 1915. Outlines the coal-tar chemical industry, the German coal-tar dyestuff industry, processes of manufacture, products, consumers, etc. Diagrams illustrate the derivation of coal-tar colors from other coal-tar products.

38. O'Neil, J. G. Raw benzol from coke-oven gas. Prog. Age, vol. 29, Oct. 16, 1911, pp. 860–861. By American improvements, all the light oil and the heavier oil vapors can be obtained from coke-oven gas, free from traces of the absorbing oil. Ninety per cent of this raw benzol will distill over at 120° C., and contain every trace of the benzene oils in the coke-oven gas. The quality of the raw benzol, whether from coal of 19 or of 30 per cent volatile matter, does not vary much , but the yield varies. English practice is poor, as only 55 to 60 per cent of the raw product distills over at 120° C. Raw benzol works well as a motor fuel, and dissolves carbon in the combustion cylinders.

39. Porter, H. C. Coal-tar products and the possibility of increasing their manufacture in the United States, with a chapter by C. G. Storm on coal-tar products used in explosives. Tech. Paper 89, Bureau of Mines, 1915, 89 pp. Contains statistics on coal-tar by-product recovery in the United States, and briefly discusses various coal-tar products. In 1913 the United States produced 150,000,000 gallons of coal tar and 7,500,000 gallons of "gas benzol." More than 100,000,000 gallons of benzol might have been obtained from the coal made into coke and gas. A table showing the fractions of average coal tar and their uses is given.

40. Puning, Franz. Benzol and its recovery from coal gas. Gas Record, vol. 7, 1915, pp. 173–179. The average benzol recovery from American coals is 2 gallons per net ton. From 1,000 cubic feet of gas produced from average coal about 0.18 gallon of benzol can be recovered. Author discusses characteristics of benzol, uses of benzol, method of recovering benzol, light-oil recovery plant, apparatus of light-oil plant, crude benzol recovery plant, purification of the crude benzol, apparatus of purifying plant, and profits of a benzol plant. Properties of "Types of benzol," including boiling points, color, reactions with sulphuric acid, and specific gravity, are included.

41. ———. Benzol—how it is recovered from coal gas. Iron Trade Rev., vol. 53, 1913, pp. 625–632, 653. Describes work done abroad in recovering benzol and what can be done here. Charts show the profits to be derived, production in European countries, proportionate constituents of light oil, boiling points and relative amounts of pure benzol in distillates, boiling points and relative amounts of toluol. Details are given of operation of a light-oil recovery plant and of a benzol rectifying and purifying plant.

42. Rau, O. Ueber die Fortschritte in der Gewinnung der Nebenprodukte beim Kokereibetriebe. Stahl und Eisen, Jahrg. 30, Bd. 2, July 20, 1910, pp. 1235–1247, 1282–1297. Reviews development of by-product coking in Europe, but contains little on benzol.

43. ROLLASON, A., and TAYLOR, A. W. The production of motor spirit from coal. Coll. Guard., vol. 106, Sept. 16, 1913, pp. 577–578. Discusses low temperature (400°–500° C.) carbonization of coal in the presence of carbon dioxide. By this process the average yield of motor spirit per ton of coal is 18 gallons. Costs and profits are shown.

44. RUMBER, —. Die wirtschaftliche Bedeutung der deutschen Benzolgewinnung, insbesondere für die Motorenindustrie. Bergbau, Jahrg. 26, 1913, pp. 689–691. Describes commercial benzol and the process of its recovery, economics of benzol production in Germany, and the use of benzol as a motor fuel. Curves show imports and exports, 1891 to 1912.

45. RUNKEL, —. Fortschritte auf dem Gebiete der Nebenproduktengewinnung. Glückauf, Jahrg. 48, Nov. 9, 1912, pp. 1833–1836. Gives a general key to the methods devised for tar recovery, washing, and purifying during 1911 and 1912. Analyses of several German coke-oven tars are included.

46. SADLER & Co. Manufacture of benzene compounds from the benzene of coal gas. Jour. Soc. Chem. Ind., vol. 4, Aug. 29, 1885, pp. 475–476. Sadler & Co. manufactured nitrobenzenes by Kendall's process, coal gas being superheated in red-hot retorts, to increase the amount of benzene, and passed into a sulphuric-nitric acid mixture. Reference is made to the processes of Leigh (1863) and of Caro (1869).

47. SAY, ADRIEN. La fabrication du coke. Paris, 1912, pp. 169–198. Describes up-to-date methods of benzol recovery, rectification, and use. The methods of Mallet, Still, Peters, are given in detail with many illustrations.

48. SCHEUR, W. Gewinnung und Verwertung von Nebenerzeugnissen bei der Verwendung von Stein- und Braunkohle. Ann. Gewerbe u. Bauwesen, Glaser, Bd. 76, June 1, 15, 1915, pp. 209–223, 239–251. Little space given to benzol, but the production of coal-tar distillates in Germany for 1911, and imports and exports for 1912, are recorded. Discusses cost of a by-product plant, the cost of production of coal-tar products in general, and the economic side of the industry in Germany.

49. SCHREIBER, F. Fortschritte in der Destillationskokerei. Ztschr. angew. Chem., Jahrg., 25, Bd. 2, Nov. 8, 1912, pp. 2289–2296. Describes the method of recovery and the uses of benzol hydrocarbons. Discusses details of the washing towers, the distillation apparatus, the purification process, and the uses of these hydrocarbons. The light oil obtained contains 55 to 60 per cent of benzol, 11 per cent of toluol, 9 per cent of xylol, 6 per cent of solvent naphtha, 10 to 20 per cent of residue boiling over 180° C. The use of dry superheated steam in working up the light oil is important.

50. SCHULZ, R. Ueber Nebenproduktengewinnung aus Generatorgas. Stahl und Eisen, Jahrg. 33, Bd. 2, July 24, 1913, pp. 1221–1225. For large central plants the Mond process is most suitable, but not when the producer is close to the furnace. The recovery of ammonia and tar from Mond gas is greater than from other recovery processes. The recovery of tar and ammonia is profitable in most cases; it does not affect the calorific power of the gas, and purified gas is less likely to foul pipes and checkerwork.

51. SIMMERSBACH, OSKAR. Benzolfabrikation. Ztschr. Ver. deut. Ing., Bd. 54, Jan. 8, 1910, pp. 70–71; Jour. Gasbel., Jahrg. 54, June 17, 1911, pp. 581–582. Gives diagram of a benzol plant, and describes washing and rectification. Discusses production and price of benzol in Germany, 1900 to 1907.

52. SIMON, H. Recent results and further development of the Simon-Carves coking process. Jour. Iron and Steel Inst., vol. 26, 1885, pt. 1, pp. 108–119. Recovery of benzol described. Preferable absorbent liquids are the heavy oils obtained by distilling tar. The scrubbers are upright cylinders, high in

proportion to their diameter, filled with pebbles or gannister. The gas flows upward to meet the descending oil. From the scrubbers, the gas passes into wash boxes also filled with heavy oil. From these wash boxes the heavy oil goes to the scrubbers, the gas containing the most benzol coming in contact with oil already charged with hydrocarbons, and the gas freest from hydrocarbons meeting fresh oil.

53. Terhaerst, Rudolf. Anlagen zur Gewinnung der Neben-Produkte im Kokereibtrieb. Stahl und Eisen, Jahrg. 18, Bd. 2, Aug. 15, 1898, pp. 747–750. After washing with water to free the gas from ammonia the gas meets a stream of coal-tar oil in the oil washers. This oil stream is as cold as possible and absorbs the benzol in the gas. The benzol is distilled later.

54. Thau, A. Über Kohlendestillation bei niedriger Temperatur. Glückauf, Jahrg. 50, May 23, 1914, pp. 834–841. Describes and compares "coalite," "Premier tarless fuel," and the Del Monte-Everett processes. Diagrams and photographs of many details are given and special attention is paid to costs. Analyses of several gas-works tars and tables of tar distillation are included.

55. Von Groeling, A. E. A new type of benzol recovery plant. Gas World, vol. 59, Sept. 6, 1913, Coke section, pp. 18–19; Chem. Abs., vol. 7, pt. 3, 1913, p. 3656. The special feature is the continuous washing plant, of which a diagram is given. The benzol is passed through 4 acid cylinders, 2 caustic soda cylinders, and 2 water cylinders. No agitating of any kind is required, and there is little loss of benzol by evaporation.

56. Von Hohenhausen, J. The separation of benzene, toluene, and xylene from crude gas-tar naphtha and crude gas benzenes. Jour. Soc. Chem. Ind., vol. 3, Feb. 29, 1884, pp. 73–77. Detailed description of nitrification test of commercial benzene and full descriptions with drawings of the Vedles toluene apparatus, the J. Aders rectifying apparatus, the Vedles rectifying system, and the Ergot system.

57. Wagner, F. H. Coal-gas residuals, benzol. 1914, pp. 121–142. Reviews benzol production, tests, properties, and uses. Describes Koppers system, Feld system, and Hirzel's methods in detail. Includes specifications and costs.

58. ——— Coal-gas residuals. Met. and Chem. Eng., vol. 12, November, 1914, pp. 696–702; Am. Gas Light Jour., vol. 101, Nov. 16, 1914, pp. 305–309. Describes Feld by-product system at length. Naphthalene, cyanogen, ammonia, and benzol are discussed in detail, and the following diagrams given: Condensing plant, Feld hot-tar washer, ammonia still, plant for extracting ammonia and hydrogen sulphide and producing ammonium sulphate. Costs, earning capacities, and yields are given.

59. Warnes, A. R. Coal tar distillation. London, 1913. Chapter entitled "Plant for the recovery of benzols, naphthas, etc." (pp. 90–112), contains a detailed description of a benzol plant, and discusses at length the distillation and rectification of benzol and naphtha.

60. Wattleworth, D. R. The production of toluene. Gas World, vol. 62, June 5, 1915, Coke section, p. 10. Coal distillation works yield a maximum of 0.3 to 0.4 gallon of toluene per ton of coal carbonized. The following synthetic reactions for increasing yield of toluene from coal-tar products are noted: Action of methyl chloride on benzene in the presence of aluminium chloride, action of methyl bromide and sodium on bromo benzene in ethereal solution, oxidation of xylene to toluic acid, and treatment of this with sodium hydroxide, distillation of cresylic acid with zinc dust. These reactions can be used if the demand for toluene becomes serious.

61. Weger, Max. Über seltenere und Reinpräparate aus Steinkohlenteer. Ztschr. angew. Chem., Jahrg. 22, Bd. 1, Feb. 19, 1909, pp. 338–346. Author discusses benzene, toluene, xylene, giving specifications for the pure products,

and a list of impurities likely to be found in them, with many literature references on each.

62. Weiss, J. M. Coal-tar light-oil in the United States. The manufacture, nature, and uses of products derived therefrom. Eighth Intern. Cong. Appl. Chem., vol. 10, 1912, pp. 287–301; Jour. Ind. Eng. Chem., vol. 5, January, 1913, pp. 61–65. General outline of the industry.

63. Wilhelm, E. Ueber die Gewinnung von Benzolprodukten. Ztschr. chem. Apparatenkunde, Jahrg. 2, Jan. 1, 15, 1907, pp. 3–10, 33–37. Discusses the practical chemistry of benzol production, the machinery employed, and methods of purification. Full description of the Gasser system. The wash oil is a mixture of equal parts of creosote oil and anthracene oil, which absorbs 2 per cent of benzol.

64. Anonymous. The Becton toluene recovery plant. Jour. Gas Lighting, vol. 129, 1915, p. 798; Jour. Soc. Chem. Ind., vol. 34, Apr. 30, 1915, p. 416. The Gas Light & Coke Co. at Becton has treated daily 15,000,000 cubic feet of gas for recovery of toluene. Research and large-scale experiments have provided information on the most suitable wash oil and the quantity needed to extract the maximum amount of toluene without reducing the calorific value or the illuminating power of the gas. By limiting the amount of oil little benzene is extracted, and the crude spirit obtained is rich in toluene, xylene, and naphthalene. The wash oil and the light spirit are separated by steam. One-third gallon of pure toluene per ton of coal carbonized is obtained.

65. ——— By-product coking and benzol recovery. Iron and Coal Trades Rev., vol. 87, 1913, pp. 652–654. Reviews a paper by G. S. Cooper before the Junior Institute of Engineers, and a pamphlet by Franz Puning, issued by the Koppers Company, of Chicago. Cooper states that the yield of by-products from 100,000 tons of a typical South Yorkshire coking coal is: Coke, 70,000 tons; tar, 5,000 tons; sulphate of ammonia, 1,500 tons; benzol, 1,000 tons; gas, 550 million cubic feet. The coking property of coal is discussed, and the essentials of good ovens are mentioned. Puning treats the recovery of benzol, toluol, xylol, and gives much valuable information on the distillation of light oil and the recovery of aromatic hydrocarbons. Products from 1,000 gallons of light oil are on an average as follows: 500 gallons of crude benzol, 120 gallons of crude toluol, 60 gallons of crude xylol, 70 gallons of solvent-naphtha. Two hundred and fifty gallons of wash oil with naphthalene remain in the still.

66. ——— A coke expert's views of the recovery of benzene, toluene, etc., from coal-gas. Jour. Gas Lighting, vol. 131, Sept. 7, 1915, pp. 502–503. The best mediums for absorption are light creosote oils boiling between 220° and 250° C. That best results may be obtained the oil should pass directly through the system against the flow of gas. In distilling it is necessary to heat the oil to 115° C. before it comes in contact with the direct steam used to vaporize the products and carry them into the condenser. The oil should be at least over 105° C.; otherwise steam is condensed and water accumulates in the oil. Many other practical details of operation are included.

67. ——— Common difficulties in by-product coke oven plants: their cause and remedy. Difficulties with benzol plants. Gas World, vol. 63, Aug. 7, 1915, Coke section, pp. 11–12. The first difficulty is that too much benzol remains in the gas after having passed the scrubbers or washers. The wash oil should be brought into intimate contact with the gas at a temperature not over 25° C.; no advantage is gained by special efforts to cool it to 15° C. There are four general types of apparatus for removing benzol: Scrubbers filled with wooden grids; rotary brush washers; baffle washers; and centrifugal washers, the first type being probably the most effective. Water may get into the wash

oil in the scrubbers or washers, in the oil coolers, in the preheaters, or in the benzol still. There is no good way of removing this water and the aim should be to prevent its getting into the oil, either by additional cooling surface for the gas or by a superheater for the steam. Discusses loss of oil, obstructions in the cooler, and lighting and fires.

68. Anonymous. The Coppée system of ammonia and benzol recovery. Gas World, vol. 59, Nov. 1, 1913, Coke section, pp. 18–19. Gives diagrams of a complete benzol-recovery plant. The gas is passed through scrubbers, where it is sprayed with creosote oil; the crude benzol obtained is treated with sulphuric acid and caustic soda and distilled.

69. ——— Direct recovery coke ovens and benzol plant at New Brancepeth Colliery. Iron and Coal Trades Rev., vol. 83, 1911, pp. 605–606. The benzol from 40 ovens is recovered with creosote oil in 4 hurdle scrubbers. The benzol is pumped through an overhead main to benzol house, where it is distilled. Not many details of actual operation are given.

70. ——— Zur Gewinnung der Nebenerzeugnisse bei der Koksfabrication. Stahl und Eisen, Jahrg. 13, Bd. 1, Jan. 15, 1893, pp. 59–65. Benzol recovery is carried out by three methods: Compressing the gas, cooling the gas to low temperature, washing the gas with a solution that absorbs benzol. General practice with the last method is outlined, and the writer emphasizes the need of considering the surface of contact of washer, so that the contact between gases and oil may be as intimate as possible. The tar oils obtained in distillation may be used for washing. The washers should be placed at considerable height. A chart of benzol prices, 1884 to 1892, is added.

71. ——— Motor spirit from coal. Science Abstracts, vol. 17, 1914, Section B, electrical engineering, pp. 298–299. Describes experimental plant at Cheswick, for working the Del Monte process, and its mechanical details. From a ton of cannel coal 17.4 gallons of motor spirit, 17.6 gallons of fuel oil, 37.5 pounds of paraffin wax, 51 pounds of pitch, and 54.5 pounds of sulphate of ammonia are obtained.

72. ——— Otto ovens and direct-recovery installation at Trimdon Grange. Gas World, vol. 61, Aug. 1, 1914, Coke section, pp. 13–14. A good diagram of Otto process for recovery of crude and refined benzol, is given, with general descriptive matter.

73. ——— Process for the recovery of toluene from coal gas. Jour. Gas Lighting, vol. 129, 1915, pp. 378–379; Jour. Soc. Chem. Ind., vol. 34, Feb. 27, 1915, p. 170. A description of Dr. Davidson's plant. Plans and working directions are given for a toluene extraction plant dealing with 800 tons of coal, or 10,000,000 cubic feet of gas per diem. Two pounds of pure toluene can be recovered from 10,000 cubic feet of coal gas. The debenzolized oil is mixed with a small quantity of benzene (4 to 5 per cent), and pumped at a rate of about 1,300 gallons an hour through scrubbers, heaters, and condensers. Commercial toluene is redistilled in a still of the Hickmann or Savalle type.

74. ——— Production of benzol. Gas World, vol. 58, 1913, p. 605. A list of plants in England producing benzol, and the number of ovens. The yield of crude benzol per oven is 80 to 90 gallons per week.

75. ——— The recovery of benzol from coke-oven gas. Coll. Guard., vol. 106, 1913, p. 1158. Describes Koppers's process. Coke-oven gas is cooled to atmospheric temperature, and passed through a series of tower scrubbers. From 1,000 gallons of crude benzol the following products are obtained: 500 gallons of 90 per cent benzol, 120 gallons of 90 per cent toluol, 60 gallons of 90 per cent xylol, 70 gallons of 90 per cent solvent naphtha. The remaining 250 gallons is used again for scrubbing.

76. Anonymous. The recovery of benzol from coke-oven gases. Chem. Eng., vol. 14, 1911, pp. 448-451. Reviews benzol-recovery industry. Describes a Still plant, and discusses each of the products: Benzol, toluol, xylol, and solvent and heavy naphthas.

77. ——— Recovery of benzol from coke-oven gases. Iron and Coal Trades Rev., vol. 75, 1907, pp. 1567-1568. Shows benefit to be derived from recovering benzol directly instead of by distillation of tar. Details given of a Still plant and of method of working, with discussion of costs.

TECHNICAL RECOVERY—PATENTS.

78. Aarts, J. G. Process and apparatus for the manufacture of coke, ammonia, illuminating gas, tar, benzene, etc., by the distillation of coal. English patent 8187, Apr. 17, 1905; French patent 353128, Apr. 7, 1905; Jour. Soc. Chem. Ind., vol. 24, Oct. 16, 1905, p. 1008. The material to be carbonized is charged continuously into a retort, passing first into hottest part in center, then into zones gradually becoming cooler. To obtain a high proportion of gas, a maximum temperature of 1,100° C. is required. For coke and by-products temperature is kept at 600° to 800° C.

79. Barbet, E., et Fils, et Cie. Apparatus for the direct extraction of benzol from heavy oils. French patent 473082, Sept. 5, 1913. Heavy oil containing 2 per cent of benzol is separated into various fractions in 2 successive operations carried out in column distilling apparatus. The oil is first distilled by heating coils supplied with high-pressure steam, and naphthalene then separated by injecting low-pressure superheated steam.

80. Davis, G. E. Improvements in the distillation of coal and in obtaining and treating products therefrom. English patent 5717, Nov. 30, 1882. The coal, previously moistened with the spent ammoniacal liquors from former operations, is heated to a comparatively low temperature, with a view to obtaining a maximum yield of benzene and a minimum yield of paraffins. The purified gases are brought into contact with a high-boiling hydrocarbon oil. The light hydrocarbons absorbed may be distilled off later.

81. ——— Obtaining benzol and other hydrocarbons. English patent 4468, Sept. 18, 1883; Jour. Soc. Chem. Ind., vol. 3, June 29, 1884, pp. 369-370. The gases containing benzene are compressed and cooled, after which they are brought in contact with a heavy hydrocarbon oil to absorb benzene and other volatile hydrocarbons. To obtain all of the benzene, the gases must be allowed to expand, and during expansion passed through washers or scrubbers containing an absorbent.

82. Gewerkschaft König Ludwig. Neurung in dem Verfahren der Cokerei mit Gewinnung von Nebenprodukten. German patent 136780, Klasse 12r., May 20, 1899. The wash oil containing benzene, toluene, xylene, and naphthalene is treated with steam superheated to 300° until the wash oil contains no more naphthalene. The regenerated wash oil is returned to the scrubbers.

83. Heinzerling, C. Process for the separation and recovery of benzol, alcohol, ether, and other easily volatile substances from air or other gas. English patent 12390, July 4, 1892; Jour. Soc. Chem. Ind., vol. 12, July 31, 1893, p. 591. The vapor-laden gas is compressed by a pressure of 2 to 6 atmospheres and cooled in a series of condensers to −60° C. to −80° C., being first cooled to 8° or 10° C., then allowed to expand in a motor cylinder where the lower temperature is reached.

84. **Heinzerling, C.** Verfahren zur Gewinnung von Benzol und dessen Homologen aus den bei der Steinkohlen- und Brandschieferdestillation resultierden Gasen. German patent 66644, Klasse 12, Oct. 21, 1891; Jour. Gasbel., Jahrg. 36, 1893, p. 480. The gases are compressed and led into solutions of strongly cooled sodium or calcium chloride or similar salts, the benzol separating on the surface of the liquid. The escaping gases are re-used to cool the salt solutions. For a modification of this process see English patent 12390, July 4, 1892. Heinzerling.

85. **Hélouïs, N. A.** An improved process for manufacturing benzene and its homologues by the distillation of coal, wood, turf, oil, and other substances. English patent 6904, June 8, 1885. The process is based on the production of acetylene throughout the process of coal distillation. The temperature in the retorts never exceeds a dull red heat; gas and superheated steam are led into retorts; a pressure of ⅛ of an atmosphere being maintained. By these methods formation of acetylene is aided, and production of more complex compounds avoided.

86. **Hirzel, Heinrich.** Abtreiben von Rohbenzol aus Waschöl. German patents 99379, Klasse 12, Dec. 22, 1896; 96228, Klasse 12, June 22, 1897; Chem. Central-Blatt, Jahrg. 70, Pt. 1, Feb. 1, 1899, p. 400. The wash oil is passed through columns at a temperature of 108° to 115°, meeting steam which removes the benzene.

87. ——— Distilling apparatus. U. S. patent 991205, May 2, 1911; Chem. Abs., vol. 5, July 10, 1911, p. 2326. Benzol recovery plant. Connections for passing wash-oil, alternately chilled and heated, through a gas washer and a still. The oil is uniformly heated in the form of a film within the still, and a part of the gas, withdrawn from the inlet to the gas washer, is circulated over the film, through the condenser and back to the inlet.

88. **Irwin, Wilfrid.** An improved still for the extraction of benzene, toluene, and xylene from their admixture with creosote or other heavy oils; English patent 8665, Apr. 13, 1898. Apparatus comprises a tower or column constructed of segments, with a dephlegmating or condensing chamber above the oil inlet. The heavier substances are condensed within the column, leaving the lighter to pass to the condenser.

89. **Kendall, J. A.** Improvements in the treatment of coal gas and similar gases in order to render the same more suitable for obtaining benzol and its derivatives therefrom. English patent 1541, Mar. 30, 1882. The gas is passed through a cast-iron or earthenware pipe heated to a bright red, in which coke or other suitable medium may be placed. Certain hydrocarbons are destroyed and the benzol content of the gas is increased.

90. **Koppers, Heinrich.** Improvements in the process of separating light oils from washing oil. English patent 15965, July 10, 1913; Gas World, vol. 60, Apr. 4, 1914, Coke section, p. 24. The heavy oil vaporized by the first distillation is redistilled.

91. ——— Verfahren zum Abtreiben von Benzol und Homologen aus Gemischen dieser Körper, Leichtöl u. dgl. mit Verwendung von Dampf als Heizmittel. German patent 277,189, Klasse 12r., Mar. 20, 1912; U. S. Patent 1,098,734, June 2, 1914. Apparatus for extraction of benzol from oil. Steam is used to produce heat, the supply of steam being regulated by the pressure of the vapor of the distillate.

92. ——— Washing out hydrocarbons from natural gas with oil. U. S. patent 1,107,803, Aug.18, 1914. The oil is put in a bell washer, which is placed in a wrought-iron jacket into which the gas is introduced so that the pressure within and without washer is equalized.

93. Kunow, Alfred. Verfahren zur Gewinnung von Leichtölen aus den Nebenprodukten der Kohlendestillation unter Anwendung von Oelen als Absorptionsmittel. German patent 143,307, Klasse 26d., July 17, 1902. The oils or their vapors are blown through a jet into gas conduit. More complete contact is thus obtained.

94. Marks, G. C. Triple-action apparatus for the recovery of benzene or other products contained in certain gases. English patent 10,577, Apr. 28, 1897; Jour. Soc. Chem. Ind., vol. 16, Sept. 30, 1897, p. 728. The gas is passed through the solvent liquid in revolving drums, ascending as small bubbles. This action is augmented by a liquid cascade or rain produced by a revolving system of buckets.

95. Mellor, Samuel. Improvements in the manufacture of benzol, nitro-benzol, nitro-toluol, and their homologues. English patent 5,604, Nov. 25, 1882. Benzol is first removed from coal gas by subjecting the gas to a pressure of four or more atmospheres in a suitable vessel, and is then nitrated with nitric acid. By a second method, the coal gas is passed through a nitrating mixture or over porous material impregnated with a nitrating mixture, and the gas deprived of benzol is washed with a milk of lime or other alkaline solution.

96. Pitt, S. Improvements in the extraction of the benzols contained in heavy oil, and in classifying or fractionating the several hydrocarbons which accompany the benzols. English patent 19,321, Sept. 10, 1898; see also Heinzerling, English patent 12,390, July 14, 1892. The heavy oils fall in layers to meet a current of steam. The light oils and higher hydrocarbons are separated, by slow condensation at definite temperatures, from the mixture of water and volatile hydrocarbons carried over by steam during distillation. From Société Anonyme pour la Construction des Appareils Savalle, Paris.

97. Plauson, H., and Schroder, P. Obtaining low-boiling hydrocarbons (benzene substitutes) from bituminous coal. English patent 4340, Feb. 19, 1914; Jour. Gas Lighting, vol. 131, Aug. 31, 1915, p. 458. One hundred parts by weight of lignite are ground with 100 parts of hydrocarbons—such as crude naphtha—and 3 to 5 per cent nitrate salts added. Air and steam at 600° C. to 700° C. are passed into a chamber, and mixture of naphtha and nitrates blown in. Four per cent of the coal and 18 per cent of crude naphtha are said to be converted into low-boiling (80° to 150° C.) constituents utilizable as benzene substitutes.

98. Schultz, G. Improved process for obtaining benzol and similar hydrocarbons. English patent 26771, Nov. 25, 1896; Chem. Central-Blatt., Jahrg. 70, Bd. 1, May 17, 1899, p. 1120; Jahrg. 71, Bd. 1, Jan. 10, 1900, p. 158. Coal-tar oils are conveyed into highly heated or glowing retorts, or hot coke ovens, preferably provided with a suction apparatus. The vapors, which contain many aromatic hydrocarbons resulting from decomposition, are condensed in the usual manner. Schultz's German patents are abstracted in the two references in Chemisches Central-Blatt.

99. Soly, A., and Davier, E. Process of recovering benzene and homologous compounds. French patent 424242, Nov. 24, 1910. The vapors are aspirated, by means of a pump, upward through a series of water columns. The benzene remains condensed on surface of water, and can be drawn off.

100. Sorel, E. Extraction of benzols and other bodies dissolved by heavy oils, and separation of their constitutents. English patent 243, Jan. 4, 1900; Jour. Soc. Chem. Ind., vol. 19, Apr. 30, 1900, p. 337. Extraction is effected by using steam in a continuous distillation column. Separation is based upon

each of the vapors forming with steam a mixture that boils at a fixed temperature. An excess of steam is mixed with these bodies and the mixture subjected to fractional cooling. Thus naphthalene, xylene, toluene, and benzene may be separated from one another.

101. STILL, C. Process for distilling the benzene hydrocarbons from saturated (gas) washing oils. English patent 16493, July 15, 1912; German patent 251,562, Klasse 12r., Aug. 9, 1911; Gas World, vol. 59, Oct. 1, 1913, Coke section. p. 21. Above the distilling column is a reflux condenser where the vapors are cooled sufficiently to condense naphthalene and the constituents of the washing oils only. The condensate is then run into another condenser, where it is cooled until no more naphthalene separates.

BENZENE, TOLUENE, AND RELATED PRODUCTS—TESTS, IMPURITIES, PROPERTIES, USES.

102. BARKER, J. T. Experimentelle Bestimmung und thermodynamische Berechnung der Dampfdrucke von Toluol, Naphthalin, und Benzol. Ztschr. phys. Chem., Bd. 71, 1910, pp. 235–253. The vapor pressure determined is as follows: Toluene at 25.8° C., 28.75 mm.; at 14.8°, 16.80; 0°, 6.86; −21°, 1.61; −78°, 0.0058. Naphthalene 20°, 0.064; 30°, 0.164; 40°, 0.378. Benzene (solid), −77.5°, 0.018. These values are compared with those calculated from Nernst's theory.

103. BAY, I. Dosage du sulfure de carbone dans les benzols. Compt. rend., t. 146, Jan. 20, 1908, p. 132. The carbon bisulphide in benzene may be determined by precipitation with phenylhydrazine, phenylhydrazine-phenylthiocarbazate being formed, and the precipitation being complete after two or three hours. The substance is collected on a tared double filter paper, washed with pure benzene, and dried. The method is extremely accurate, the greatest error being less than 0.5 per cent of the amount determined. The values are generally too high.

104. BIEHRINGER, JOACHIM. Beiträge zur Kenntniss der leichtest flüchtigen Antheile des Steinkohlentheers. Dingl. poly. Jour., Bd. 276, 1890, pp. 78–90. The author discusses carbon bisulphide and various methods for its determination and removal.

105. BRUNI, G. Über das Gefrieren von gemischen stellungsisomerer Benzolderivate. Ztschr. Elektrochem., Jahrg. 16, May 1, 1910, p. 285. Bruni states that the conclusions reached in Fischer's experiments exactly agree with his own earlier results. In general, isomerides in the benzene series do not form solid solutions, but substances having analogous substituting groups in the same positions do.

106. BURRELL, G. A., and ROBERTSON, I. W. The determination of benzol in gas mixtures. Jour. Ind. Eng. Chem., vol. 7, August, 1915, pp. 669–670. The same method is used as in separating illuminants. The percentage of benzol was checked by combustion analysis and it was found that all of the benzol was separated at −78° C.

107. ——— The separation of the illuminants in mixed coal and water gas. Jour. Ind. Eng. Chem., vol. 7, January, 1915, pp. 17–21. A method for determination of benzene is described as follows: The gas, freed from water vapor, is introduced into a liquefaction bulb and cooled to −78° C. After removal of as much gas as possible with a pump, the condensed liquid is vaporized, and its pressure read on a manometer. This pressure compared with the original pressure of the gas gives the percentage of benzene.

108. CHURCH, S. R. Methods for testing coal tar and refined tars, oils, and pitches derived therefrom. Jour. Ind. Eng. Chem., vol. 3, April, 1911, pp. 227–233.

Describes tests, standardized by a manufacturing company, for examining raw materials and products of tar distillation. Methods for distilling and for determining specific gravity of light oils and benzols are given.

109. Church, S. R., and Weiss, J. M. Paraffin bodies in coal-tar creosote and their bearing on specifications. Jour. Ind. Eng. Chem., vol. 6, May, 1914, pp. 396–398. Furnishes a key to literature on the dimethyl-sulphate method for separating aromatic from open-chain compounds. The authors believe the method is of little value.

110. Colman, H. G. Determination of the percentage of toluene in commercial solvent naphtha. Jour. Gas Lighting, vol. 129, 1915, pp. 314–315. One hundred c. c. of the sample is distilled at rate of one drop per second, and distillate up to 138° C. is collected. If this does not amount to 35 c. c., a second 100 c. c. is fractionated, and the distillates combined. If combined distillates do not amount to 35 c. c., sample is considered to be free from toluene. Otherwise 35 c. c. of distillate is mixed with 50 c. c. of pure toluene and 15 c. c. of pure benzene, toluene in this mixture determined as previously described, and the toluene content of solvent naphtha calculated.

111. ——— Determination of the percentage of toluene in commercial toluol. Jour. Gas Lighting, vol. 129, 1915, pp. 196–198; Jour. Soc. Chem. Ind., vol. 34, Feb. 27, 1915, pp. 168–170. From a large number of distillations of mixtures of known amounts of benzene, toluene, xylene, an analytical process has been devised. One hundred c. c. of sample is distilled under standard conditions, and from amounts passing over below 105° C., and above 117° C., amount of pure toluene present can be calculated by aid of a table. Specific details are given.

112. Davis, G. E. On the valuation of the crude naphthas of commerce. Jour. Soc. Chem. Ind., vol. 4, Nov. 30, 1885, pp. 645–649. A lengthy discussion of the distillation of commercial benzol, and the estimation of benzene, toluene, and xylene.

113. Deniges, Georges. Dosage volumétrique rapide du thiophéne dans la benzine. Bull. Soc. chim. France, ser. 3, t. 13, 1895, pp. 537–546; t. 15, 1896, pp. 1064–1065; Jour. Soc. Chem. Ind. vol. 15, Oct. 31, 1896, p. 746, vol. 14, Oct. 31, 1895, p. 893; Jour. Chem. Soc., vol. 68, 1895, A. 1, p. 411; A. 2, p. 372. Thiophen is estimated, by precipitating, as a basic thiophen mercuric sulphate, with an excess of mercuric sulphate solution and titrating back with decinormal silver nitrate solution until permanent opalescence caused by the separation of benzene appears.

114. Dieterich, Karl. Analysis and valuation of motor fuels; fourteen methods for examining them. Automobile, vol. 33, July 29, August 5, 1915, pp. 202–205, 247–249, 267. Properties of benzol and gasoline are compared, with use of these fuels discussed. Describes methods of testing as follows: Specific gravity, color and other external characteristics, test of odor, timing of evaporation in a clock crystal, behavior toward litmus, color reaction with sulphuric acid, benzol test with isatine-sulphuric acid, benzol test by nitration, test with "Dracorubin" paper, nitrate of silver test, calcium carbide test for water, fractional distillation, refractometer test, mentions comparative prices.

115. Ellerton, J. G. The estimation and elimination of sulphur compounds in commercial benzol. Jour. Soc. Chem. Ind., vol. 31, Jan. 15, 1912, pp. 10–12. Discusses various methods of estimating carbon bisulphide and thiophen in benzol. The Nickels method was adopted for carbon bisulphide, and the Deniges method for thiophen. Carbon bisulphide may be separated by fractional distillation, but removal of thiophen requires treatment with the strongest sulphuric acid, which involves a loss of 8 to 12 per cent of the

benzol. Experiments with ozone, hydrogen peroxide, and lead oxide for removing thiophen were not satisfactory.

116. Forel, S. Ueber die Destillation von technisch reinem Benzol und seinen Homologen, sowie von technischen Benzolen (90-proc. u. 50-proc.) und einigen Mischungen von Benzol, Toluol, und Xylol. Chem. Ztg., Jahrg. 21, May 19, 1897, pp. 393–394. Describes distillation flask and column. Tables show results of distilling mixtures of benzol, toluol, and xylol.

117. Frank, Fritz. Commercial benzol. Gas World, vol. 36, 1902, pp. 182–184; Jour. Gas Lighting, vol. 78, Aug. 6, 1901, pp. 341–342. Reviews history of benzol, and describes uses and tests (boiling point, specific gravity, carbon bisulphide, thiophen, paraffin hydrocarbons, olefins, resinifiable unsaturated compounds, smell, kindling point). German prices and imports during the period 1890 to 1900, are given. From Chem. Ind., 1901.

118. —— Der Handelsverkehr mit Benzolen, ihre Zusammensetzung, Untersuchung und Verwerthung. Chem. Ind., Jahrg 24, Apr. 15, May 1, 1901, pp. 237–241, 262–266. Reviews early history of benzol industry and literature on determination of boiling point; gives details and a drawing of Bannow's distillation apparatus as modified by Kraemer and Spilker; also directions for taking specific gravity, determining carbon bisulphide by Liebermann and Seyewetz method, determining paraffins according to Lunge's method, finding amount of resinous and unsaturated bodies, and for determining individual benzene homologues, in commercial benzol. States uses.

119. Friswell, R. J. An improved method of purifying toluene, benzene, etc. Chem. News., vol. 68, July 21, 1893, p. 27; Jour. Soc. Chem. Ind., vol. 12, July 31, 1893, pp. 589–590. Process intended for purifying products obtained in making oil-gas. Purification of toluol by agitation with concentrated or fuming sulphuric acid is not recommended. Crude toluene boiling between 110° and 130° C. should be agitated thoroughly for 4 hours with 10 per cent by volume of sulphuric acid, specific gravity 1.803, then drawn off, washed once with enough caustic soda to remove traces of the acid, and rectified. Loss should not exceed 5 per cent.

120. Hailstone, H. J. Estimation of sulphur in coal gas and benzol. Gas World, vol. 58, 1913, p. 37. Products of combustion of either gas or benzol are led through a solution containing hydrogen peroxide in the presence of ammonia. Resulting sulphur trioxide is precipitated as barium sulphate.

121. Haissig, Max. Ueber Dichten von Benzol und Toluol-Mischungen. Chem. Ztg., Jahrg. 21, Nov. 10, 1897, p. 939. A table shows specific gravity at 17.5° C. of mixtures of benzene and toluene from 100 per cent benzene to 100 per cent toluene, at differences in content of 2½ per cent.

122. Haller, Albin, and Michel, Edmond. Sur un procédé de purification, au moyen du chlorure d'aluminium, de carbures renfermant du thiophène, ses homologues et d'autres impuretés. Application à la purification du benzéne et du toluéne. Bull. Soc. chim. France, t. 15, ser. 3, Sept. 5, 1896, pp. 1065–1070. Aluminium chloride may be used to purify large quantities of benzene. The hydrocarbon is agitated with aluminium chloride, after a time decanted from viscous liquid produced, and then distilled in the absence of water. Benzene which has been purified with sulphuric acid requires about 1 per cent of aluminium chloride; but crude hydrocarbon boiling at 80 to 81° may require as much as 5 per cent. Process is applicable to toluene, but results are less satisfactory.

123. Harding, E. P., and Doran, J. Quantitative determination of carbon bisulphide in a mixture of benzene and carbon bisulphide. Jour. Am. Chem. Soc., vol. 29, October, 1907, pp. 1476–1480. Author reviews literature; his method depends on formation of potassium xanthate, by a potassium hydrox-

ide solution from mixtures of benzene and carbon bisulphide. The potassium xanthate is titrated with an excess of standard cupric acetate solution, and the excess of cupric acetate determined by sodium thiosulphate.

124. Henriques, R. Quantitative Trennung von Steinkohlentheer-und Petroleum-Kohlenwasserstoffen. Chem. Ztg., Jahrg. 19, May 22, 1895, pp. 958–959. Coal-tar hydrocarbons may be separated from petroleum hydrocarbons by treating a mixture of the two with twice its volume of sulphuric acid containing 5 per cent of sulphur trioxide. The coal-tar hydrocarbons are entirely dissolved.

125. Heusler, F. Beiträge zur Chemie des Braunkohlentheers. Ber. Deut. chem. Gesell., Jahrg. 25, Bd. 2, 1892, pp. 1665–1678; 28, Bd. 1, 1895, pp. 488–498. Discusses oxidation of coal-tar hydrocarbons by potassium permanganate in alcoholic solution in the cold (G. Wagner's method of separation); method of separation by fractional bromination; and action of sulphuric acid of various strengths on hydrocarbons. Benzene and toluene were estimated by formation of dinitro products. Methods are given for proving presence of paraffins, terpenes, indene, cumarone, and naphthalene. A detailed analysis of the fraction of coal-tar boiling below 180° is included.

126. ——— Über die Entfernung des Thiophens aus dem Benzol mittels Aluminiumchlorid. Ztschr. angew. Chem., Jahrg. 9, Dec. 15, 1896, p. 750; Jour. Chem. Soc., vol. 72, 1897, pt. 1, p. 402. Thiophen was removed from benzol by boiling 9 hours in a reflux apparatus with aluminum chloride. (5.8 g. of aluminium chloride to 120 c. c. of benzol.) The distillate was washed with caustic soda and dried with calcium chloride.

127. Holde, David. Zur Unterscheidung von Benzol und Erdölbenzin. Jahrb. Chemischen Technologie, Jahrg. 41, 1896, pp. 554–555. Gives directions for distinguishing between benzol and benzine through the different solubility of asphalt in these products.

128. Hubbard, Prevost. Coke-oven tars of the United States. Office of Public Roads Circular 97. 1912, 11 pp. Contains a well-tabulated series of analyses of coke-oven tars obtained from representative manufacturers throughout the United States. Light oils run from 0.3 per cent to 2.5 per cent, most tars giving between 1 and 2 per cent.

129. Irwin, Wilfrid. A new test for sulphur in benzol for use in gas works. Jour. Soc. Chem. Ind., vol. 20, May 31, 1901, pp. 440–442. (*See also* Lunge, Georg, Coal-tar and ammonia, 1909, p. 774.) Discusses sulphur in benzol, and ordinary tests for it. Proposes a new method, an adaption of the barium sulphate method of testing for sulphur in gas. Specifications for commercial benzol are given.

130. Jacobsen, Oscar. Ueber die Entstehung der Benzolkohlenwasserstoffe bei der trocknen Destillation. Ber. Deut. chem. Gesell., Jahrg. 10, Bd. 1, 1877, pp. 853–861; 1009–1015. Explains formation of benzene, toluene, xylene, mesitylene by condensation of simpler hydrocarbons formed during heating, and describes his researches on phoron-cumol, and on the recovery of xylenes from tar-oil.

131. Johnson, E. S. Determination of carbon disulphide and total sulphur in commercial benzene. Jour. Am. Chem. Soc., vol. 28, August, 1906, pp. 1209–1220; Jour. Soc. Chem. Ind., vol. 25, Oct. 15, 1906, pp. 955–956. Estimate carbon bisulphide by converting by potassium hydroxide into potassium xanthate, precipitating with copper sulphate, and weighing as copper oxide. Estimate sulphur by converting with a stream of oxygen into sulphuric acid and weighing as sulphate.

132. Krieger, A. Benzolbestimmung im Gase. Jour. Gasbel., Jahrg. 58, Feb. 6, 1915, pp. 61–64; Chem. Abs., vol. 9, Aug. 10, 1915, pp. 2141–2142; Am. Gas

Light Jour., vol. 102, 1915, pp. 299–300; Jour. Gas Lighting, vol. 129, 1915, p. 450; Jour. Soc. Chem. Ind., vol. 34, Mar. 31, 1915, p. 266. Summarizes earlier work on benzol determination. Author modifies Müller's method of washing gas with cooled paraffin oil, and makes it available for coke-oven gas by using improved absorption apparatus. Benzol is absorbed almost quantitatively. Details of method are given.

133. LUNGE, GEORG. Coal tar and ammonia, 1909. Chapter entitled "Benzene—physical and chemical properties" (pp. 177–183) is valuable for its long list of references. Chapter entitled "Toluene—physical and chemical properties" (pp. 183–184) also contains references. In chapter entitled "Theory of the formation of benzene hydrocarbons in coal tar" (pp. 241–245) the author discusses formation of benzene from acetylene and other hydrocarbons and gives many references to the literature.

134. LUNGE, GEORG, AND BERL, ERNST. Chemisch-technische Untersuchungsmethoden. Berlin, 1911, Bd. 3. In the chapter on "Bestimmung des Benzoldampfes" (pp. 275–281) the Deville method of freezing out benzene from gaseous mixtures at −22° C., and the Harbeck and Lunge method, as modified by Lunge, of determining benzene as dinitrobenzene, are described in detail. Many references to literature. Chapter entitled "Untersuchung der Handelsbenzole" (pp. 407–423) gives distillation tables and methods of distilling. Literature on analytical methods for carbon bisulphide, for thiophene, for paraffin hydrocarbons, and for unsaturated and resinous compounds is reviewed. Shows methods of distinguishing between petroleum and coal-tar products. Chapter entitled "Untersuchung des Leichtöls" (pp. 391–397) contains many references and describes properties of light oil; analytical methods are given for determining specific gravity, boiling point, amount of phenols and pyridine bases present, and for running distillation tests. Tables of distillation tests of mixtures of benzol and toluol are included.

135. LUNGE, GEORG, AND VON KÉLER, H. Untersuchung Zweier Rohbenzole aus Koksofengasen. Ztschr. angew. Chem., Jahrg. 7, Nov. 1, 1894, pp. 637–640; Jour. Soc. Chem. Ind., vol. 14, Feb. 28, 1895, p. 143; Mineral Ind., vol. 5, 1896, p. 189. Distinguishes two varieties of crude benzol—absorption benzol and compression benzol—discusses chemical composition, physical properties and tests, and gives tables of distillations of these products. A 90 per cent benzol from Upper Silesia was examined by distillation. Composition by weight of benzols: Absorption benzol, 85.10 per cent benzene, 11.63 toluene, 1.54 xylene; compression benzol, 67.03 per cent benzene, 15.61 toluene, 2.18 xylene. All other constituents are given.

136. MARKS, ARTHUR. Benzol as a motor fuel; impurity troubles. Gas World, vol. 62, May 1, 1915, Coke section, p. 15. Tests for carbon bisulphide and sulphuric acid are important. Sulphuric acid may be neutralized by mixing benzol with ½ to ⅓ its amount of petrol, which is generally alkaline. When higher fractions boiling over 150° C. are allowed to pass over in distillation of light oil, the benzol may contain cumarone, which is extremely objectionable and not readily detected.

137. MASTBAUM, H. Benzin und Benzol. Ztschr. angew. Chem., Jahrg. 22, Jan. 29, 1909, p. 202. The French nomenclature is followed in Portugal for differentiating the petroleum from the coal-tar distillate, the first being "ether de petroleo" and the second "benzina," "benzenio," or "benzol."

138. NORTHALL-LAURIE, D. A new method for the determination of toluene in commercial toluols. Analyst, vol. 40, September, 1915, pp. 384–389. Mixtures of benzene and toluene, toluene and xylene, of known composition, by volume, were prepared and their boiling points determined. By plotting the boiling points against percentages a curve was drawn, from which the

composition by volume for all boiling points could be read. Detailed directions for working and full description of apparatus are included. More than 50 per cent of toluene must be present or toluene must be added in known amount to the sample.

139. Reeve, C. S., and Lewis, R. H. Application of the dimethyl sulphate test for determining small amounts of petroleum or asphalt products in tars. Jour. Ind. Eng. Chem., vol. 5, April, 1913, pp. 293-295. Reviews literature on this test for separating aromatic from open-chain hydrocarbons.

140. Rittman, W. F., and Egloff, Gustav. Physical constants of gas oils and derived tars. Jour. Ind. Eng. Chem., vol. 7, June, 1915, pp. 481-484. Gives fractional distillation, specific gravity, surface tension, and refractive index of 4 gas oils and the derived tars. Difference between constituents of an aliphatic oil and of its derived tar are made evident by differences between volatility-gravity, volatility-refractive index, and volatility-surface tension relations. Aromatic hydrocarbons can be formed from the aliphatic hydrocarbons in petroleum. Various physical constants of typical gas oils and of derived tars have been tabulated.

141. Schelenz, Hermann. Benzin und Benzol. Ztschr. angew. Chem., Jahrg. 21, Dec. 18, 1908, pp. 2577-2579. Discusses various names, past and present, used to denote coal-tar fraction and petroleum fraction.

142. Schenk, Konrad. Über die Bestimmung des Gesamt-Schwefels im Handelsbenzol. Chem. Ztg., Jahrg. 38, Jan. 17, 1914, pp. 83-84; Gas World, vol. 60, 1914, p. 490. The benzol is burned in a specially constructed apparatus, sulphur dioxide absorbed in a 1 per cent hydrogen peroxide solution, and sulphuric acid estimated by titration with decinormal alkali, and methyl-orange as an indicator.

143. Schwalbe, Carl. Sulphur content of pure benzols. Jour. Soc. Chem. Ind., vol. 24, Mar. 31, 1905, p. 271; from Ztschr. Farben-u Textil-Ind., Jahrg. 4, 1905, pp. 113-119. Presents a table showing sulphur content of several commercial benzols and discusses various methods of testing.

144. ——— Verfahren zur Ausscheidung der Schwefelverbindungen, insbesondere des Schwefelkohlenstoffs und Thiophens aus Theeröldestillaten. German patent 133,761 Klasse 12o., Dec. 10, 1901. Oil to be purified is shaken with ammonia in gas or liquid form. The benzol when removed from the oily residue formed is free from carbon bisulphide.

145. Smith, J. K. Imperishable color-standard solutions, and their application to the classification of benzol washing tests. Jour. Soc. Chem. Ind., vol. 18, Mar. 31, 1899, p. 212. Many practical observations on commercial testing of benzol. Three tests are discussed in detail; specific gravity test, behavior of benzol on distillation, and behavior with acid.

146. Société Anonyme de St. Denis. Verfahren zum Ausscheiden des Thiophens aus dem Rohbenzol. German patent 79,505, Klasse 12, July 27, 1894. Crude benzol is heated several hours with 0.5 per cent of aluminium chloride, then decanted, distilled, and washed with a concentrated solution of sodium carbonate. The process depends on aluminium chloride forming condensation products with thiophen more easily than with benzene hydrocarbons.

147. Stavorinus, D. Zur Bestimmung des Schwefelkohlenstoffs im Benzol. Jour. Gasbel., Jahrg., 49, Jan. 6, 1906, p. 8; Jour. Soc. Chem. Ind., vol. 25, Feb. 15, 1906, p. 139. Twenty-five c. c. of sample are mixed with 70 c. c. of 96 per cent alcohol and 10 c. c. of twice-normal sodium hydroxide. After half an hour 5 c. c. of concentrated hydrogen peroxide is added, the alcohol removed by evaporation, and the sulphate estimated.

148. Thorpe, Edward. Dictionary of applied chemistry; benzene. Vol. 1, 1912, pp. 439–447. Surveys chemical and physical properties of benzene and its homologues, methods of preparation, purification, and nitration.

149. Valenta, E. Über die Verwendung von Dimethyl-sulfat zum Nachweis and zur Bestimmung von Teerölen in Gemischen mit Harzölen und Mineralölen und dessen Verhalten gegen fette Öle, Terpentinöl und Pinolin. Chem. Ztg., Jahrg. 30, Mar. 1906, pp. 266–267. Dimethyl-sulphate dissolves true tar oils, leaving any mineral oil or resin oil. An accurately measured amount of oil should be shaken with twice its quantity of dimethyl-sulphate.

150. Votocek, E., and Potmesil, R. Qualitative Bestimmung von Schwefelkohlenstoff in technischen Benzolen. Chem. Repertorium, suppl. to Chem. Ztg., Jahrg. 25, Sept. 21, 1901, p. 275. This rapid method depends on Hofmann's reaction between carbon bisulphide and aniline. The alkaline sulphide formed is detected by the nitroprusside reaction.

151. Weiss, J. M. Coal-tar light oil in the United States; the manufacture, nature, and uses of products derived therefrom. Jour. Ind. Eng. Chem., vol. 5, January, 1913, pp. 61–65. Describes in detail method of distilling light oil and the uses of benzols in rubber, paint, and varnish, lacquers, and imitation lacquers, and miscellaneous industries. Gives table of properties of benzols. Discusses method of working up and uses of carbolic acid. Production figures are for 1910–1911.

152. ——— Commercial benzol in the United States. Jour. Ind. Eng. Chem., vol. 3, January, 1911, pp. 10–13. Briefly discusses manufacture and uses of benzol. Outlines commercial tests. Commercial benzol is a mixture of benzene, toluene, xylene in proportion varying according to the purity of benzol.

153. ——— Determination of carbon bisulphide in benzol. Jour. Ind. Eng. Chem., vol. 1, August, 1909, pp. 604–605. Describes in detail method based on conversion of carbon bisulphide into potassium xanthate, the oxidation of latter by bromine in alkaline solution, and precipitation of sulphur as barium sulphate.

154. White, A. H., and Hess, W. A. Composition of some coal tars from American gasworks. Jour. Soc. Chem. Ind., vol. 19, June 30, 1900, pp. 509–510. Amount of light oil by weight ran from 0.49 per cent to 3.2 per cent. Authors state that American tars examined are inferior to German tars and are not well adapted to by-product recovery. Results are tabulated.

155. Young, Sydney, and Fortey, Emily. The vapor pressures and boiling points of mixed liquids. Jour. Chem. Soc., vol. 83, pt. 1, 1903, pp. 45–77. On page 59 is a table of boiling points of mixtures of benzene and toluene for pressures of 660 to 830 mm., and for 24.94,50.00, and 72.46 per cent of toluene.

156. Anonymous. Distinguishing between petroleum benzine and coal-tar benzene. Jour. Soc. Chem. Ind., vol. 15, Jan. 31, 1896, p. 53, from Mitt. k. k. Versuchsanst., Bd. 13, 1895, p. 241. Mixtures of petroleum benzine and coal-tar benzene may be analyzed by relative solubility of asphalt in them. As an empirical test this method is satisfactory.

157. ——— The estimation of benzol. Jour. Gas Lighting, vol. 131, July 27, 1915, p. 180. A measured volume of coke-oven gas is passed through nitro-benzene or creosote oil. The product is distilled up to 120° C. (distilling to 135° C. is suggested), and from the benzol obtained the number of gallons of benzol per ton of coal may be calculated.

158. ——— The Marchal apparatus for estimating the value of by-products in fuel. Coll. Guard., vol. 95, May 8, 1908, pp. 881–882. Illustrates an apparatus by which coal may be carbonized on a small scale and yield of by-products obtained automatically in a series of washing flasks.

159. Anonymous. War Office benzol test. Gas World, vol. 62, Feb. 6, 1915, Coke section, p. 21. Gives detailed directions for distillation test of benzol. In Great Britain a permit to sell must be obtained from the High Explosives Committee if 95 per cent of the benzol does not distill over below 90° C.

PRODUCTION OF GASOLINE AND AROMATIC HYDROCARBONS FROM PETROLEUM.

160. Adiasiewitsch, A. W. Verfahren zur Behandlung von Erdölen und Bitumen behufs Gewinnung von festen Harzen und aromatischen Kohlenwasserstoffen. Chem. Central-Blatt, Jahrg. 67, Jan. 22, 1896, p. 401; from Journal of the Russian Physical-Chemical Society. Deoxygenized air is blown through oil residues at 150° to 200°, while the oil is subjected to electric discharges.

161. Armstrong, H. E. On the manufacture of gas from oil. 1. Preliminary note on certain by-products of the manufacture of gas from oil by the Pintsch process in relation to the question of the conditions under which benzenoid hydrocarbons are formed. Jour. Soc. Chem. Ind., vol. 3, Sept. 29, 1884, pp. 462–468. Naphthenes, and olefins from ethylene to monylene, and also crotonylene were found among the products of cracking.

162. Armstrong, H. E., and Miller, A. K. On the decomposition and genesis of hydrocarbons at high temperatures—The products of the manufacture of gas from petroleum. Jour. Chem. Soc., vol. 49, 1886, pp. 74–93; Chem. News, vol. 51, June 26, 1885, pp. 307–308. Describes methods of separation and chemical behavior of the hydrocarbons formed. In manufacture of oil gas benzene, toluene, and other aromatic hydrocarbons were obtained by compression. The liquid deposited during decomposition of gases at 10 atmospheres pressure was a complex mixture of hydrocarbons rich in aromatic compounds.

163. Boissieu, M. de. Pyrogenation appliquée aux résidus de pétroles russes. Bull. Soc. chim. France, t. 64, 1893, pp. 2–3. By dry distillation the residue known as mazóutt yielded 12 to 15 per cent of aromatic hydrocarbons, and might have yielded more by quick lowering of temperature or by pressure. Most of the crude benzol distilled between 100° and 140°. The amounts of benzol, toluol, and xylol were 8.04, 2.57, and 1.05 per cent.

164. Dvorkovitz, Paul. Distillation, cracking, and gasification of petroleum hydrocarbons. Petroleum Rev., vol. 17, Sept. 28, 1907, pp. 176–179. Discusses action of heat on petroleum hydrocarbons below and above their boiling points, and gives practical applications. Illustrates an apparatus for manuufacturing aromatic hydrocarbons from petroleum.

165. ——— Die Vergasung von Kohle und flüssigen Kohlenwasserstoffen. Jour. Gasbel., Jahrg. 37, 1894, pp. 10–11; Jour. Soc. Chem. Ind., vol. 12, May 31, 1893, pp. 403–412. Describes manufacture of gas from coal and from oil. Oil vapors were led over hot plates at 1,300° (704° C.) to 1,600° F. (871° C.), and a large amount of aromatic hydrocarbons obtained.

166. Edeleanu, L. Die Petroleumindustrie in Rumänien und die Chemie des rumänischen Petroleums. Chem. Rev. Fett-Harz-Ind., Jahrg. 8, 1901, pp. 63–66. Discusses possibility of obtaining benzene and toluene from petroleum by superheating. By superheating a fraction obtained at 70° to 170° yielded 30 per cent of aromatic hydrocarbons.

167. ——— Refining petroleum by liquefied sulphur dioxide. Bull. Am. Inst. Min. Eng., No. 93, Sept., 1914, pp. 2313–2332. If petroleum distillate is agitated with liquid sulphur dioxide at low temperatures aromatic hydrocarbons are dissolved, but paraffins and naphthenes are unaffected. Thus a distillate may be obtained practically free from aromatic constituents.

168. Engler, C., and von Höfer, H. Das Erdöl, Bd. 1, 1913. Chapter entitled "Darstellung von aromatischen Kohlenwasserstoffen aus Erdöl und Erdölrückständen," pp. 587–593, is extremely useful for its survey of European literature on aromatic hydrocarbons from petroleum, which contains many abstracts of papers not easily obtained in America.

169. Faraday, Michael. On new compounds of carbon and hydrogen, and on certain other products obtained during the decomposition of oil by heat. Philos. Trans. Roy. Soc. London, vol. 115, 1825, pp. 440–466. Of great historical interest, as Faraday was the first investigator. Describes a new compound, termed "Bicarburet of hydrogen" but now known as "benzol." This compound was found in tar from oil gas made from fish or vegetable oil.

170. Letny, A. Ueber die Einwirkung hoher Temperatur auf Petroleum, Braunkohlentheer und andere ähnliche Stoffe. Dingl. poly. Jour., Bd. 229, 1878, pp. 353–365. Light oils were obtained from heavy petroleum by conducting it through a tube heated to not over 300° C. The tar contained many aromatic hydrocarbons. A good article with careful tests and much data.

171. Lewes, V. B. The production of oil gas from Russian petroleum. Jour. Soc. Chem. Ind., vol. 11, July 30, 1892, pp. 584–590. Discusses formation of benzene in cracking oil at temperatures of 500° to 1,000° C. Three methods of benzene formation are given: Polymerization of acetylene, decomposition of higher paraffins into benzene and hydrogen, and decomposition of naphthenes. Amount of unsaturated hydrocarbons decrease with higher temperature; saturated and acetylene compounds increase. At high temperature the unsaturated split off saturated compounds.

172. Nikiforoff, A. Gewinnung von Benzol aus Erdöl und Erdölrückstanden. Chem. Ztg., Jahrg. 20, Bd. 1, Jan. 1, 1896, p. 8. The hydrocarbons boiling below 200° C. are first distilled off, and remainder subjected to a second distillation under 2 atmospheres pressure at high temperature. Yield of benzol is 12 per cent of weight of oil used.

173. Ogloblin, W. N. Die Darstellung von Benzol und seiner Homologen aus russischem Naphta nach dem Verfahren A. N. Nikiforoff. Chem. Central-Blatt, Jahrg. 75, Bd. 2, Sept. 14, 1904, p. 830; from Ztschr. Farb.-Textil-Chem. Describes Nikiforoff's process. Production of aromatic hydrocarbons is easiest from crude petroleum. Method of consecutive decomposition results in dehydrogenation of naphthenes, and their transformation into aromatic hydrocarbons. Describes details of obtaining tar, and of its fractional distillation. Benzol and toluol obtained from petroleum, according to results of N. D. Zelinski are free from thiophene and its homologues, dehydro and tetra-hydro compounds. Absence of sulphur compounds, bases, phenols, etc., makes the use of alkaline and acid purification agents unnecessary.

174. ——— Preparation of benzene and its homologues from Russian petroleum. Jour. Soc. Chem. Ind., vol. 23, Oct. 15, 1904, p. 931; from Ztschr. Farb. Textil-Chem. Nikiforoff's method. Petroleum is first heated to 525° to 550° C. in iron retorts fitted with iron channels through which petroleum flows. The portion obtained boiling below 200° C. is again distilled under 2 atmospheres pressure at 700° to 1,200° C., and fraction boiling up to 120° C., which is called "gray benzol," amounts to 12 per cent of original petroleum. Fraction obtained at 120° to 200° C. is again decomposed under 2 atmospheres pressure.

175. Pamfilow, S. M. Gewinnung von Leuchtgas und aromatischen Kohlewasserstoffen aus Erdöl. Chem. Ztg., Jahrg. 21, Dec. 15, 1897, p. 1069. Petroleum vapor was passed through a coiled tube at continually increasing tempera-

ture below that required for oil gas. A tar containing 15 per cent of benzol was obtained.

176. Redwood, Boverton. The Russian petroleum industry. Jour. Soc. Chem. Ind., vol. 4, Feb. 28, 1885, pp. 70–81. Astatki is decomposed in a cupola regenerative furnace patented by Nobel, and benzene, anthracene, and naphthalene obtained by two distillations. Yields of tar and of hydrocarbons are given.

177. Rudnew, W. Ueber die Produkte der Destillation des Petroleumgastheers. Dingl. poly. Jour., Bd. 239, 1881, pp. 72–74. Ten to twelve per cent of benzol, also some toluol and naphthalene, were obtained from tar formed in making oil gas.

178. Veith, Alex. Gewinnung aromatischer Körper aus den Rückständen. Bolley and Wiedemann. Handbuch der chemischen Technologie; Erdöl., 1862, p. 435. Petroleum residues are allowed to flow through red-hot iron tubes at 700° to 800°. On an average, 17 per cent of benzol is obtained, but yield of aromatic hydrocarbons depends on temperature and on regularity of feeding. Coal-tar gives about 8 per cent of benzol.

179. Williams, Greville. Fluid hydrocarbons obtained by the compression of petroleum gas. Chem. News, vol. 49, May 2, 1884, p. 197. When the gas obtained from petroleum at high temperature is compressed, as in Pintsch system, a liquid containing benzene, toluene, and olefins is obtained. Describes the method of separation of olefins from benzene.

180. Zelinsky, N. D. Ueber die Darstellung aromatischen Kohlenwasserstoffe aus dem Erdöl. Chem. Ztg., Jahrg.26, Jan. 22, 1902, p. 68. Describes consecutive decomposition under increasing pressure. The aromatic fraction passing over between 75° and 180° amounts to 14.1 per cent of the petroleum, and contains 58 per cent of benzol and 28 per cent of toluol. Zelinsky has repeated Nikiforoff's experiments on a larger scale.

181. Anonymous. Neue Gewinnungsmethode von Benzol, Naphthalin und Anthracen. Dingl. poly. Jour., Bd. 246, 1882, pp. 429–432. Gives many references to early literature on production of aromatic hydrocarbons from petroleum and from coal tar. Is valuable as a key to the work of the time.

PATENTS ON THE PRODUCTION OF AROMATIC HYDROCARBONS FROM PETROLEUM (AND TAR OILS).

182. Abel, C. D. Improvements in the treatment of hydrocarbons for their purification and conversion into other products. English patent 4,769, 1877; Chemische Ind., Jahrg. 1, 1878, p. 411. Patent covers a process devised by C. Friedel and J. M. Crafts. Hydrocarbons are treated with aluminum chloride or other metallic chlorides at temperatures of 100° to 600° C. Metallic oxides also may be added. Petroleum is converted into light oils; naphthalene forms benzene and toluene.

183. Dvorkovitz, Paul. Manufacture of gas and by-products from liquid hydrocarbons. English patents 7,983, Apr. 27; 19,641; 21,647, Nov. 26, 1892. Process relates to manufacture of benzol and toluol by internal heating of the fixing chamber for gasifying the hydrocarbons. Steam is decomposed to produce water-gas, which passes into gasifier, where liquid hydrocarbon is gasified. The water-gas is there carbureted, and passes into the fixing chamber, where benzol and toluol are deposited.

184. ——— Treatment of liquid hydrocarbons, and the production therefrom of gas and valuable by-products. English patent 23,671, Dec. 5, 1894; Jour. Soc. Chem. Ind., vol. 15, Feb. 29, 1896, p. 104. Petroleum or other liquid hydrocarbon flows through an extensive series of retorts in which it is progressively

subjected to temperatures ranging from 300° C. to 1,000° C. The retorts are of great width and of small depth and are arranged in inversely inclined positions, connecting pipes being heated by hot gases so that the lowest retorts rise to a temperature of 1,000° C., while upper retorts are at 300° C.

185. Dvorkovitz, Paul. Verfahren und Apparat zur Gewinnung von Gas und werthvollen Nebenprodukten aus flüssigen Kohlenwasserstoffen. German patent 88,122, Klasse 26, Apr. 7, 1895; Ber. Deut. chem. Gesell., Jahrg. 29, Bd. 4, 1896, p. 930. A zigzag furnace in which oil runs down over flat plates. the lower plates being at higher temperatures, up to 1,000° C. and the upper plates at 300° C. The oil may be warmed beforehand by passing it through heated tubes.

186. Hausman, J., and Pilat, S. Verfahren zur Darstellung aromatischer Kohlenwasserstoffe durch Einleiten von Erdöl oder Erdölfraktionen in Dampfform in erhizte Röhren über Kontaktsubstanzen. German patent 227,178, Klasse 12o, Apr. 1, 1909. Petroleum vapor is passed into a heated tube containing a contact substance consisting of an oxide or peroxide or salt of some metal, and capable of acting as an oxygen carrier. Iron, lead, cerium oxides, or manganese peroxide, iron sulphate, or calcium manganite may be used.

187. Hirzel, Heinrich. Verfahren und Apparat zur Destillation und gleichzeitigen Reinigung von Petroleum, Theer und Theerölen, Harz und Harzölen u. dgl. auch zur Zersetzung derselben und gleichzeitigen Reinigung der Zersetzungsprodukte. German patent 34,315, Klasse 23, Apr. 21, 1885; Ber. Deut. chem. Gesell., Jahrg. 19, Bd. 3, 1886, p. 121. Aromatic hydrocarbons were formed by passing petroleum vapors through a container filled with porous material, such as wood carbon, heated to glowing. See German patent 45,769, Klasse 26, 1888, for further description of the apparatus.

188. Hlawaty, F. Verfahren zur Gewinnung von Benzol, Toluol, Xylol, usw. aus Petroleum, Petroleumrückständen, Steinkohlentheer, Steinkohlentheeröl usw. German patent 51,553, Klasse 22, Aug. 11, 1888; Ber. Deut. chem. Gesell., Jahrg. 23, Bd. 5, 1890, p. 438. Sawdust, or any form of cellulose, caustic alkalies, and petroleum were mixed, and led with a current of steam superheated to 400° through a glowing iron tube filled with coke or other contact substance. Condensed vapor furnished benzene, toluene, and other aromatic hydrocarbons. Yield of aromatic compounds may be increased by leading back uncondensible gases into the vessel containing the petroleum and cellulose.

189. Hlawaty, F., and Friedmann, E. Improvements in the process of preparing benzol and its homologues, naphthalene, and anthracene from petroleum, petroleum scales, tar, tar oils, paraffin, vaseline, ozokerite, and the like, and from natural illuminating gas. English patent 2242, Feb. 11, 1890. The vapors are led through red-hot pipes filled with coke or pumice.

190. Holcgreber, J. Process for obtaining benzene from petroleum or its distillates. English patent 17272, July 28, 1913; French patent 460,827, July 28, 1913; Jour. Soc. Chem. Ind., vol. 33, Jan. 15, Aug. 31, 1914, pp. 17, 825. Petroleum vapors mixed with hydrogen are passed through metal or other tubes containing catalytic substances at temperatures of 180° to 300° C. Metals are used as catalyzers.

191. Meffert, F. Verfahren zur Herstellung von aromatischen Kohlenwasserstoffe und von Ammoniak. German patent 99,254, Klasse 12, 1897; Chem. Central-Blatt, Jahrg. 70, Bd. 1, Jan. 11, 1899, p. 159. Oil and superheated steam are led together into a retort filled with some refractory material at 1,200° to 1,400°. The condensed products contain benzene.

192. Meikle, J. Improvements in obtaining benzene, toluene, and other products from mineral oils and liquid tars. English patent 23,649, Oct. 24, 1896; Jour. Soc. Chem. Ind., vol. 16, Nov. 30, 1897, p. 905. The oil is injected by aid of superheated steam into a retort filled with coke or other carbonaceous material at red heat. Benzene is separated from resulting gaseous mixture by condensation.

193. Nikiforoff, A. Apparat zum Zerlegen der flüssigen Kohlenwasserstoffe zwecks Benzol Gewinnung. German patent 85,884, Klasse 12, Mar. 12, 1895. A pressure of 2 atmospheres is used in the retort. To obtain certain advantages from receiver being at atmospheric pressure, a small opening, kept from stopping by a rod with a fine point, connects retort and receiver.

194. ——— Improvements in the manufacture of benzene, anthracene, naphthalene, and other products from naphtha and naphtha residues. Russian patent 290, Dec. 31, 1894; English patent 10957, Aug. 27, 1886; Jour. Soc. Chem. Ind., vol. 6, Aug. 31, 1887, p. 540; Engler und Höfer, Das Erdöl, Bd. 1, 1913, pp. 588–589; German patent 143,549, Klasse 12r., Oct. 10, 1901. The oil is first decomposed in cylindrical retorts at 515° to 550° C. at ordinary pressure to obtain hydrocarbons boiling below 200° C. and having a specific gravity of 0.800 to 0.810. These are distilled again, and then decomposed at 700° C. to 1,200° C. and 2 atmospheres' pressure, when they are transformed into hydrocarbons of the aromatic series. In general, 12 per cent of weight of oil is obtained as benzene, ½ per cent as anthracene, 2 per cent as naphthalene. The German patent describes apparatus for purifying distillation products from decomposition of petroleum or coal tar.

195. ——— Improved method and apparatus for producing benzenes and their homologues. U. S. patent 755,309, 1904; French patent 315,428, Oct. 29, 1901; Jour. Soc. Chem. Ind., vol. 21, Apr. 30, 1902, p. 541. Liquid hydrocarbons boiling below 200° C. are subjected to double decomposition under pressure at two different temperatures (about 525° and 900° C.). The primary decomposition products are washed before passing them through condenser. In order to increase working area of secondary retorts they are surmounted by a third. Internal pressure is increased by means of a perforated plug in discharge pipe of retorts, and rakes are provided for cleaning. Aromatic compounds are extracted by solvents. See English patent 17,450 of 1907.

196. ——— Verfahren und Vorrichtung zur Gewinnung von aromatischen Kohlenwasserstoffen aus Naphta; German patent 224,070, Klasse 12 o, Mar. 24, 1908; Chem. Abs., vol. 4, pt. 3, Nov. 20, 1910, p. 3297. In producing aromatic hydrocarbons from naphtha the hydrocarbons obtained by coking the naphtha are washed, then atomized in an atmosphere of an inactive gas at 800° C. to 1,000° C., the necessary high temperature being maintained by introducing additional water gas. Benzol may be obtained up to 20 per cent.

197. Williams, Greville. Process for the separation of benzene and its homologues from the liquid obtained by compression of petroleum gas. English patent 4,663, Mar. 10, 1884. The liquid is treated with dilute nitric acid or alkaline nitrate, and distilled.

ACTION OF HEAT ON HYDROCARBONS—CONSTITUTION OF PETROLEUM.

198. Armstrong, H. E., and Miller, A. K. On the decomposition and genesis of hydrocarbons at high temperatures. 1. The products of the manufacture of gas from petroleum. Jour. Chem. Soc., vol. 49, 1886, pp. 74–93; Chem. News, vol. 51, June 26, 1885, pp. 307–308. Treats of methods of separation and chemical behavior of various hydrocarbons formed. Benzene, toluene, and other aromatic hydrocarbons were obtained by compression of oil gas.

199. Armstrong, H. E., and Miller, A. K. On the effect of high temperatures on petroleum hydrocarbons. Chem. News, vol. 49, June 20, 1884, pp. 285–286. Briefly describes decomposition of paraffin; an extension of work of Thorpe and Young (Proc. Roy. Soc. London, vol. 20, 1871–1872, pp. 488–491; vol. 21, 1872–1873, pp. 184–201).

200. Berthelot, M. Action de la chaleur sur quelques carbures d'hydrogéne. Théorie des corps progénes. Ann. chim. phys., ser. 4, t. 9, 1866, pp. 445–483. Investigation of action of heat on acetylene, and formation of benzene.

201. ——— Des carbures pyrogénés. Ann. chim. phys., ser. 4, t. 12, 1867, pp. 5–96; t. 16, 1869, pp. 143–187. A detailed account of action of heat on hydrocarbons, formation of polymers, synthesis of benzene hydrocarbons.

202. Bone, W. A., and Coward, H. F. Thermal decomposition of hydrocarbons. Pt. 1. Methane, ethane, ethylene, and acetylene. Jour. Chem. Soc., vol. 93, 1908, pp. 1197–1225. Temperature in experiments ranged from 500° to 1,200°. Special attention was paid to the time factor. At low temperatures acetylene shows a tendency to polymerize, forming benzene hydrocarbons. Polymerization reaches a maximum between 600° and 700° C.

203. Day, D. T. On changes effected by heat in constitution of ethylene. Am. Chem. Jour., vol. 8, June, 1886, pp. 153–169. Ethylene begins to change at 350° C. At 400° C. it is decomposed with formation of marsh gas, ethane, and liquid products.

204. Engler, C. Die Zersetzung hochmolekularer Kohlenwasserstoffe durch mässige Hitze. Ber. Deut. chem. Gesell., Jahrg. 30, Bd. 3, 1897, pp. 2908–2920. An investigation of the chemical substances formed by the decomposition of petroleum through heat, with special reference to products of the cracking process.

205. Engler, C., and Halmai, B. Über Naphthenbildung; Die Produkte der Druckerhitzung eines Zylinderöls. Ber. Deut. chem. Gesell., Jahrg. 43, Bd. 1, 1910, pp. 397–405. Oil was heated to 430° or 470° C. under a pressure as high as 128 atmospheres. Chemistry of products obtained is discussed.

206. Engler, C., and Jezioranski, L. Ein Beitrag zur Kenntniss der elementaren Zusammensetzung der Erdöle. Ber. Deut. chem. Gesell., Jahrg. 28, Bd. 3, 1895, pp. 2501–2505. By distillation at 200° to 250° a fraction is obtained that becomes richer in unsaturated hydrocarbons on redistillation.

207. Engler, C., and Routala, O. Über Naphthenbildung im Erdöl. Ber. Deut chem. Gesell., Jahrg. 42, Bd. 4, 1909, pp. 4610–4631. Chem. Abs., vol. 4, Mar. 20, 1910, pp. 766–767. Discusses formation of naphthenes from olefins by heating under pressure. Heavy viscous lubricating oil, formed by the action of aluminium chloride on amylene, was heated under pressure. Low-boiling oils were obtained.

208. Gustavson, G. Zerfall der Kohlenwasserstoffe des amerikanischen und kaukasischen Petroleums bei niedrigen Temperaturen. Chem. Central-Blatt, Jahrg. 53 (12), June 28, 1881, p. 353. Action of aluminium bromide and hydrobromic acid on these petroleums, formation of Al $Br_3C_4H_8$.

209. Haber, F. Theorie der pyrogenen Reactionen aliphatischer Kohlenwasserstoffe. Ber. Deut. chem. Gesell., Jahrg. 29, Bd. 3, 1896, pp. 2691–2700. Describes investigations on polymerization of hydrocarbons at 600° to 1,000°, with special attention to the thermodynamics of the decomposition of aliphatic hydrocarbons by heat.

210. Haber, F., Samoylowicz, H., and Oechelhaeuser, H. Ueber die Zersetzung von Hexan und Trimethyläthylen in der Hitze. Jour. Gasbel., Jahrg. 39, June 13, 20, July 4, 11, Dec. 5, 12, 19, 1896, pp. 377–382, 395–399, 435–438, 452–455, 799–805, 813–818, 830–833; Jour. Chem. Soc., vol. 72, pt. 1, 1897, pp.

305–308. At temperatures up to 900° benzene is formed by polymerization of acetylene. Over 900°, the benzene formed begins to decompose. At 606° hexane decomposes considerably, olefins, paraffins, hydrogen, and benzene being formed.

211. KRÄMER, G., AND BÖTTCHER, W. Ueber die Beziehungen des Erdöls zu den Kohlenwasserstoffen der Braun-und Steinkohlentheeröle. Ber Deut. chem. Gesell., Jahrg. 20, Bd. 1, 1887, pp. 595–609. Aromatic hydrocarbons can be formed from aliphatic hydrocarbons by superheating, or by heating under pressure. Naphthenes were not formed from paraffin hydrocarbons. A fraction of petroleum 150° to 160° C. was passed after extracting all unsaturated compounds, through a red hot tube. A distillate containing 7.5 per cent of unsaturated compounds was obtained.

212. LEWES, V. B. The action of heat upon ethylene. Proc. Roy. Soc. London, vol. 55, 1894, pp. 90–107, vol. 57, 1894/1895, pp. 394–404. Chem. News, vol. 69, Feb. 23, Mar. 2, 9, 16, 1894, pp. 87–89, 103–105, 111–112, 125–127; vol. 71, June 7, 14, 21, 1895, pp. 273, 286–287, 298–299. Ethylene is converted by heat into acetylene and methane. Acetylene is at once polymerized into secondary products, or decomposed into carbon and hydrogen. Initial rate of decomposition is extremely rapid, but increase in rate of flow diminishes amount of decomposition when heated area is small, the effect being less with increased length of flow.

213. MABERY, C. F. Constituents of Pennsylvania, Ohio, and Canadian petroleums between 150° and 200° C. Am. Chem. Jour., vol. 19, June, 1897, pp. 419–482. Hydrocarbons found in petroleum.

214. MABERY, C. F., AND QUAYLE, W. O. Sulphur compounds and unsaturated hydrocarbons in Canadian petroleum. Am. Chem. Jour., vol. 35, May, 1906, pp. 404–432. A continuation of author's work on composition of petroleum.

215. McKEE, G. W. On the decomposition of benzene at high temperatures. Jour. Soc. Chem. Ind., vol. 23, Apr. 30, 1904, pp. 403–404. Presents diagram of apparatus employed and details of its management. Benzene begins to decompose at about 700° C., so that in adding enriching material to water gas it is not advisable to allow temperature to rise much over 700° C.

216. MARKOWNIKOFF, W., AND SPADY, J. Zur Konstitution der Kohlenwasserstoffe C_nH_{2n}, des kaukasischen Petroleums. Ber. Deut. chem. Gesell., Jahrg. 20, Bd. 2, 1887, pp. 1850–1853. Sulphur at 210° to 220° C. used to remove hydrogen. Octanaphthene decomposed to form xylene.

217. MEYER, RICHARD, AND TANZEN, AUGUST. Pyrogene Acetylen-Kondensationen Ber. Deut. chem. Gesell., Jahrg. 45, Bd. 2, 1912, pp. 1609–1633; Jahrg. 46, Bd. 3, 1913, pp. 3183–3199; Chem. Abs., vol. 6, pt. 2, Sept. 10, 1912, p. 2618; vol. 8, pt. 1, Jan. 10, 1914, p. 342. Acetylene and an equal volume of hydrogen were passed through an apparatus consisting of two tubes, one at 640° to 650°, the other at about 800°. Sixty per cent of a light-brown aromatic tar was obtained. Most suitable temperature for the formation of benzene hydrocarbons is about 650° C.

218. MEYER, R., AND FRICKE, H. Pyrogene Acetylen-Kondensationen. Ber. Deut. chem. Gesell., Jahrg. 47, Bd. 3, 1914, pp. 2765–2774; Ztschr. angew. Chem., Jahrg. 28, Jan. 29, 1915, Referate, p. 58. A systematic investigation of acetylene-tar, in which 23 compounds present in ordinary coal tar have been detected.

219. NORTON, L. M., AND ANDREWS, C. W. Action of heat on liquid paraffins. Am. Chem. Jour., vol. 8, March, 1886, pp. 1–9. Discusses action of heat on hexane, isohexane, normal pentane, and the decomposition products and temperature of decomposition.

220. Norton, L. M., and Noyes, A. A. On the action of heat upon ethylene. Am. Chem. Jour., vol. 8, March, 1886, pp. 362–364. Products formed by passing ethylene through a red-hot tube were examined. Formation of crotonylene.

221. Phillips, W. B. Fuel oil in the Southwest, with a bibliography of fuel oil generally. Trans. Am. Inst. Min. Eng., vol. 48, 1914, pp. 565–612. Bibliography contains references to the composition of petroleum.

222. Prunier, M. L. Les carbures incompletes qui prennent naissance dans le traitment pyrogéné des pétroles d'Amérique. Ann. chim. phys., ser. 5, t. 17, 1879, pp. 1–62. A study of the unsaturated hydrocarbons formed in decomposing petroleum vapor by heat. Synthesis of unsaturated hydrocarbons. Temperatures of 350° to 450° C. were used.

223. Richardson, Clifford. The petroleums of North America. A comparison of the character of those of the older and newer fields. Jour. Franklin Inst., vol. 162, July, August, 1906, pp. 57–70, 81–128. Treats of the physical properties of the petroleums and discusses the hydrocarbons in them. Many references to literature.

224. Routala, O. Ueber die Bildung der Naphthene in Erdöl. Petroleum, Jahrg. 5, Dec. 15, 1909–1910, pp. 321–322. Naphthenes similar to those found in petroleum were obtained from amylene by heating under pressure.

225. Willstätter, Richard, and Hatt, Daniel. Abbau des Cyclo-hexans zum Benzol. Ber. Deut. chem. Gesell., Jahrg. 45, Bd. 2, 1912, pp. 1464–1471. Describes the authors' methods for carrying out synthesis of benzene from cyclohexane.

226. Wischin, Rudolf. Die cyklischen Polymethylene des Erdöls. Chem. Ztg., Jahrg. 23, Bd. 2, Oct. 28, 1899, pp. 916–926; Jahrg. 28, Bd. 2, Aug. 31, 1904, pp. 814–818; Jahrg. 29, Bd. 2, Oct. 25, 1905, pp. 1126,1127. Summarizes fully the existing knowledge of these compounds, and has a good bibliography.

227. Worstall, R. A., and Burwell, A. W. Decomposition of heptane and octane at high temperatures. Am. Chem. Jour., vol. 19, December, 1897, pp. 815–845. Hydrocarbons, under similar conditions of temperature, yield same products. In these experiments crude heptane and octane were decomposed at 900°. Products tabulated. Benzol 12.5 per cent and toluol 3.0 per cent by weight were obtained.

ACTION OF CATALYZERS.

228. Aschan, Ossian. Zur Genese der Naphthene und der Naphthensäuren. Liebig's Annalen, Bd. 324, 1902, pp. 1–39; Chem. Central-Blatt, Jahrg. 73, Bd. 2, Oct. 1, 1902, pp. 895–897. At low temperature in the presence of aluminium chloride, olefins are polymerized to naphthenes.

229. Engler, C. Die Bildung der Hauptbestandteile des Erdöls. Petroleum, Jahrg. 7, Jan. 17, 1911-1912, pp. 399–403; Chem. Abs., vol. 6, May 10, 1912, pp. 1221, 1222. Discusses the influence of catalyzers, as clay, siliceous earth, sand, fullers earth, and metallic oxides, in formation of petroleum from fat at high temperatures and pressures. Proposes a scheme for characterization and division of products of various phases of bitumination.

230. Engler, C., and Severin, E. Katalyse und Erdölbildung. Ztschr. angew. Chem., Jahrg. 25, Bd. 1, Jan. 26, 1912, pp. 153–158. Largely bibliographical, and furnishes a good survey of literature dealing with action of catalyzers on petroleum.

231. Heusler, F. Über die Einwirkung von Aluminiunchlorid auf Teer-und Erdöldestillate und über die Theorie der Erdölbildung. Ztschr. angew. Chem., Jahrg. 9, May 15, 1896, pp. 288–292; Chem. Central-Blatt, Jahrg. 67, 1896, p. 144. By influence of aluminium chloride, unsaturated hydrocarbons are changed to saturated compounds, and sulphur is removed.

232. Ipatiew, W. Polymerisation der Äthylen-Kohlenwasserstoffe bei hohen Temperaturen und Drucken. Ber. Deut. chem. Gesell., Jahrg. 44, Bd. 3. 1911, pp. 2978–2987; Chem. Abs. vol. 6, pt. 1, Mar. 10, 1912, p. 736; Chem. Zentralb., Jahrg. 82, pt. 4, Dec. 13, 1911, pp. 1777–1779. Under a pressure of 70 atmospheres, polymerization of ethylene begins at 325°, and is very fast at 380° to 400°. Products obtained and mechanism of reactions are discussed. Polymerization was also carried out in presence of alumina.

233. ——— Rôle of the oxides in catalytic phenomena. Chem. Abs., vol. 2, pt. 2, July 20, 1908, pp. 1958–1959; from a paper before the Russian Physical Chemical Society, on use of nickel oxides as catalyzers. Ipatiew has published many papers on catalytic reactions at high temperatures and pressures, most of them in Ber. Deut. chem. Gesell., 1900–1915.

234. Ipatiew, W., and Dowgelewitsch, N. Zersetzung des Hexans und Hexamethylens; Isomerisation des Hexamethylens. Ber. Deut. chem. Gesell., Jahrg. 44, Bd. 3, 1911, pp. 2987–2992; Chem. Abs. vol. 6, pt. 1, Mar. 10, 1912, p. 736; Chem. Zentralb., Jahrg. 82, pt. 4, Dec. 13, 1911, pp. 1779–1780. Discusses products obtained by passing hexane and hexamethylene under ordinary pressure through an iron tube at 650° to 700° with, or without catalyzers, those used being alumina, zinc dust, and reduced copper.

235. Ipatiew, W., and Routala, O. Polymerisation des Äthylens bei hoher Temperatur und Druck in Gegenwart von Katalysatorem. Ber. Deut. chem. Gesell., Jahrg. 46, Bd. 2, 1913, pp. 1748–1755. Chem. Abs., vol. 7, pt. 3, Nov. 20, 1913, p. 3972. Chem. Zentralb., Jahrg. 84, Bd. 3, July 16, 1913, p. 234. Ethylene under 70 atmospheres pressure in presence of $ZnCl_2$ begins to polymerize at 275°. With aluminium chloride polymerization takes place at lower temperatures. Amount of naphthenes in the products increased with temperature, but was far smaller when using aluminium chloride.

236. Kusnetzow, M. Die Zersetzung der gasförmigen Kohlenwasserstoffe beim Glühen mit feinverteiltem Aluminium. Ber. Deut. chem. Gesell., Jahrg. 40, Bd. 3, 1907, pp. 2871–2873. Methane, ethane, ethylene, acetylene were decomposed into their elements.

237. Letny, A. Ueber die Zersetzung des Petroleums und des Braunkohlentheers durch Ueberhitzen. Ber. Deut. chem. Gesell., Jahrg. 11, Bd. 1, 1878, pp. 1210–1212; Chem. News, vol. 38, Oct. 25, 1878, p. 211. Petroleum vapor was conducted through glowing iron tubes, 7 feet long and 1 foot in diameter, filled with wood charcoal or coke. Tar obtained yielded about 10 per cent of benzene, toluene, and other aromatic hydrocarbons. Yield of tar was increased by using platinized carbon.

238. Liebermann, C., and Burg, O. Ueber die Zersetzung des Braunkohlentheeröls in der Glühhitze. Ber. Deut. chem. Gesell., Jahrg. 11, Bd. 1, 1878, pp. 723–726. Coal-tar oils and petroleum were passed over heated coke in a brass tube. Benzene, toluene, and anthracene were produced. (See Liebermann, C., Dingl. poly. Jour., Bd. 246, 1882, pp. 429–432.) General review of literature on new methods of obtaining benzol, naphthalene, anthracene.

239. Mailhe, A. Molekulare Spaltungen mittels fein zerteilter Metalle. Chem. Ztg., Jahrg. 32, Mar. 4, 7, 1908, pp. 229–230, 244–245; Chem. Abs. vol. 2, pt. 2, June 20, 1908, p. 1688. The author used finely divided nickel, copper, iron, or cobalt at high temperatures to perform molecular cleavages. Above 250° benzene becomes methane; cyclohexane vapors break up into benzene and methane. Origin of petroleum and action of finely divided metals on acetylene are discussed.

240. Niviere, Jean. La catalyse en chimie organique. Rev. gen. Chimie, t. 16, 1913, pp. 189–196, 220–224. Describes action of various catalytic agents for

hydrogenation, dehydrogenation, and oxidation. References furnish a key to literature of catalysis.

241. Ostromisslenski, I., and Burshanadse, J. [Über die pyrogene Zersetzung der Naphtha in Gegenwart von Katalysatoren. 1. Üeber den Bildungsmechanisums des Benzols bei der pyrogenen Zersetzung der Naphtha.] Jour. Russ. Phys. Chem. Gesell., Bd. 42, 1910, pp. 195–207; Chem. Zentralb., Jahrg. 81, Bd. 1, May 11, 1910, pp. 1711–1712; Jour. Soc. Chem. Ind., vol. 29, June 15, 1910, p. 682. Aromatic hydrocarbons are formed at high temperatures from petroleum by polymerization of acetylene, and use of catalyzers increases output. After passing petroleum vapor through a combustion tube at 700° C., containing a spiral of iron wire gauze, products contained 10.9 per cent of benzene compared with 7.5 per cent when no iron gauze was used. Using nickel instead of iron no benzene was obtained. Part 2. Description of hydrocarbons into carbon and hydrogen by the contact process; use of nickel and nickel oxides.

242. Rosenfeld, J. Zerfallsprodukte des Petroleums. Oesterr. Chem. tech. Ztg., Bd. 27, 1909, p. 1; Engler-Höfer, Das Erdöl, Bd. 1, 1913, p. 591. Describes the hydrocarbons found in tar obtained by passing petroleum through a red-hot tube. Benzene was found in first fraction under 200°. Coke or copper used as a catalyzer increased output.

243. Sabatier, Paul. Hydrogénations et déshydrogénations par catalyse. Ber. Deut. chem. Gesell., Jahrg. 44, Bd. 2, 1911, pp. 1984–2001. Methods devised by Sabatier and his students. General action of metals used as catalyzers, and their special action on many hydrocarbons.

244. Sabatier, Paul, and Senderens, J. B. Action de divers métaux divisés, platine, cobalt, fer sur l'acétylène et sur l'éthylène; Compt. rend., t. 131, July 25, 1900, pp. 267–270; Jour. Chem. Soc., vol. 78, pt. 1, 1900, p. 534. Above 200°, with iron as catalyzer; chief products in addition to hydrogen and carbon were olefins and benzene. Use of cobalt resulted in less benzene.

245. ——— Action du nickel réduit sur l'acétyléne. Compt. rend., t. 131, July 16, 1900, pp. 187–190; Jour. Chem. Soc., vol. 78, pt. 1, 1900, p. 471. A rapid current of acetylene passed over reduced nickel at a temperature above 180° gave 10.3 per cent of benzene and its homologues, 51.4 per cent of hydrogen, 36.3 per cent of ethane, 2 per cent of ethylene, and traces of acetylene. The liquid product was a mixture of aromatic and unsaturated hydrocarbons boiling between 50° and 180° C.

246. ——— Hydrogénation de l'acétylène en présence du nickel. Compt. rend., t. 128, May 8, 1899, pp. 1173–1176; Jour. Chem. Soc., vol. 76, 1899, p. 555. Mixtures of acetylene and hydrogen were passed over finely divided nickel. Main reaction was formation of ethane; but as proportion of acetylene in original mixture was increased, amount of benzene hydrocarbons in product also increased. Iron, cobalt, and copper acted similarly.

247. ——— Hydrogénation de l' acétylène et de l'ethylène en présence du platine divisé. Compt. rend., t. 131, July 2, 1900, pp. 40–42; Jour. Chem. Soc., vol. 78, pt. 1, 1900, p. 471. At 180° in presence of platinum black, condensation of products yields liquid hydrocarbons.

248. ——— Nouvelles méthodes générales d'hydrogénation. Ann. chim. phys., ser. 8, t. 4, 1905, pp. 319–480. Catalyzers, as finely divided nickel, were used. At 16° in a tube 30 cm. long benzene vapor is hydrogenized by nickel to cyclohexane. Time of reaction a factor (p. 360). At 270° to 280° cyclohexane in contact with finely divided nickel decomposes into benzene (p. 363). Reactions with homologues of benzene, using other catalysts, follow (p. 364).

249. Salzmann, M., and Wichelhaus, H. Ueber die Herstellung von Benzol aus Braunkohlentheeröl. Ber. Deut. chem. Gesell., Jahrg. 11, Bd. 2, 1878, pp. 1431–1434. Oil was passed over pumice stone, wood and animal carbon, or platinized asbestos in a tube heated to redness. Results are tabulated.

250. Smith, Clarence, and Lewcock, William. Pyrogenic decompositions—benzene. Jour. Chem. Soc., vol. 101, pt. 2, 1912, pp. 1453–1459. Describes the influence of temperature and continued heat on formation of diphenyl from benzene. Benzene was passed through an iron tube at 600° to 800° C., and results with BaO$_2$, Al$_2$O$_3$, ZnO, and Pb$_3$O$_4$ as catalyzer are tabulated. Yield of diphenyl was 10 to 59 per cent.

251. Ubbelohde, Leo, Woronin, S., and St. Philippide, —. Ueber die Einwirkung von Katalysatoren auf Erdöl. Petroleum, Jahrg. 7, Oct. 4, 1911, Jan 3, Aug. 21, 1912, pp. 9–13, 334–339, 1233–1238; Chem. Abs., vol. 7, pt. 1, Jan. 10, 1913, pp. 246–247. Porous earthenware on which nickel had been reduced in metallic form was impregnated with petroleum oil, and heated in a current of purified nitrogen or hydrogen at regulated temperatures from 180° to 500°. Various proportions of hydrogen, methane, and ethane were produced. The effect of fuller's earth, unburned kaolin, ignited alumina, and nickel upon petroleum at temperatures of 200° to 400° was studied, and a table of results and curves given, pp. 1234–1238.

252. Vohl, H. Ueber die Versetzung der Schweren Mineralöle aus Braunkohlen, Torf, bituminösen Schiefern, Petroleum, und zur Erzeugung leichterer (wasserstoffreicherer) Beleuchtungsöle. Dingl. poly. Jour., Bd. 177, 1865, pp. 58–76. Heavy oil was distilled with concentrated sulphuric acid, with sodium hydroxide, and over glowing lime. The vapors were cracked in iron tubes filled with iron filings or limestone.

253. Zelinsky, N. D. Über Dehydrogenisation durch Katalyse. Ber. Deut. chem. Gesell., Jahrg. 44, Bd. 3, 1911, pp. 3121–3125; Chem. Zentralb., Jahrg. 82, Bd. 4, Dec. 13, 1911, p. 1794. Hexamethylene hydrocarbons are easily dehydrogenated by passing them over palladium black at 300°. Dehydrogenation begins at 170° and goes on energetically at 200°. Benzol and toluol are obtained. Copper, silver, and magnesium are inactive as catalyzers.

254. ——— Über die selektive Dehydrogenisations-Katalyse. Ber. Deut. chem. Gesell., Jahrg. 45, Bd. 3, 1912, pp. 3678–3682; Chem. Abs., vol. 7, pt. 2, July 10, 1913, p. 2224. Hexamethylene hydrocarbons are readily dehydrogenated in the presence of platinum or palladium at 300°, but pentamethylene derivatives remain unchanged below 300°.

CRACKING, ACTION OF HEAT OR OF HEAT AND PRESSURE, ON PETROLEUM.

255. Armstrong, H. E. On the manufacture of gas from oil; Preliminary note on certain by-products of the manufacture of gas from oil by the Pintsch process in relation to the question of the conditions under which benzenoid hydrocarbons are formed. Jour. Soc. Chem. Ind., vol. 3, Sept. 29, 1884, pp. 462–468. Remarks on chemical products formed by cracking oil. Naphthenes and olefins from ethylene to monylene, and crotonylene were among products.

256. Bjerregaard, A. P. Studies on the pressure distillation of petroleum hydrocarbons. Jour. Ind. Eng. Chem., vol. 7, July, 1915, pp. 573–577; Pet. Rev., vol. 33, Aug. 14, 1915, p. 126. An investigation for developing on a commercial scale a process for making light-gravity hydrocarbons from the heavier fractions of petroleum. Oil is forced through a coil under pressure of 2,000 pounds per square inch.

257. Boleg, F. Apparat und Verfahren zur Destillation von Kohlenwasserstoffen. Oelen und Fetten unter Hochdruck. Chem. Rev. Fett-Harz-Ind., Jahrg. 5, 1898, pp. 9–12, 24–27. Ten to twelve per cent more of petroleum products was obtained by use of 3 to 6 atmospheres pressure when distilling.

258. Boys, Vernon. Production of motor spirit from heavy oil. Met. and Chem. Eng., vol. 12, March, 1914, p. 180. The heavy oil was subjected to the action of steam at 315°.

259. Brooks, B. T., Bacon, R. F., and Others. The preparation of gasoline and kerosene from heavier hydrocarbons. Jour. Ind. Eng. Chem., vol. 7, March, 1915, pp. 180–185. General review of the subject, with references.

260. Charitschkoff, K. W. Destruktive Destillation der russischen Erdöle. Chem. Rev. Fett-Harz-Ind., Jahrg. 17, 1910, p. 33. Results obtained by distilling at 400° (cracking) Baku and Grosny oils from which the light oils up to 270° had been removed. At a temperature a little over 400° decomposition goes on without great evolution of gas. Table of results given.

261. ——— Die Zersetzungsprodukte der Naphtha von Baku und Grosny. Chem. Ztg., Jahrg. 19, Nov. 20, 1895, p. 2083; Chem. Repertorium, Jahrg. 20, Feb. 8, 1896, p. 21; Jahrg. 26, Jan. 4, 1902, p. 7. Decomposition of all petroleums begins at 250° to 275°. Cracking can be continued till the tarry state is reached.

262. Claudy, C. H. The Burton process of "cracking" to make gasoline. Sci. Am., vol. 112, Jan. 2, 1915, pp. 5, 32. A popular article on this method.

263. Dvorkovitz, Paul. Distillation, cracking, and gasification of petroleum hydrocarbons. Petroleum Rev., vol. 17, 1907, pp. 176–179. Action of heat on petroleum hydrocarbons above and below their boiling points. Practical applications.

264. Dyer, E. J. The cracking of oils. Min. and Sci, Press, vol. 110, 1915, p. 717; Chem. Abs., vol. 9, July 10, 1915, p. 1841. Discusses briefly cracking of oils in "concentrates." It is better in general to adhere to ordinary distillation methods, because of objectionable odor and color of cracked distillates from presence of olefins.

265. Engler, C., and Halmai, B. Über Naphthenbildung; Die Produkte der Druckerhitzung eines Zylinderöls. Ber. Deut. chem. Gesell., Jahrg. 43, Bd. 1, 1910, pp. 397–405. Oil heated to 430° to 470° under a pressure of 128 atmospheres. Discusses chemistry of products obtained. Heavy mineral oils can be changed into more liquid products without formation of much gas or coke.

266. Engler, C., and von Höfer, H. Das Erdöl, Bd. 1, 1913. The chapter entitled "Der Krak-prozess" (pp. 562–578), gives a survey of European literature. Many references and abstracts of papers not easily obtained in America are given.

267. Freud, B. B. The "cracking" of hydrocarbons. Chem. Eng., vol. 21, 1915, pp. 160–162. Describes most recent processes of cracking.

268. Hall, W. A. The "cracking" of oils, with a view to obtaining motor spirit and other products. Petroleum World, vol. 12, 1915, pp. 137–145; Petroleum Rev., vol. 32, Sept. 11, 1915, pp. 207–208; Engineering, vol. 99, 1915, pp. 250–251; Mining Jour., vol. 108, Mar. 27, 1915, pp. 217–218; vol. 109, Apr. 3, 1915, pp. 227–228; Jour. Gas Lighting, vol. 129, 1915, p. 455. Oil is fed at rate of over 70 gallons per hour through inch tubes more than 300 feet in length, under 50 to 75 pounds pressure. Speed of vapor through tubes more than 5,000 feet per minute; temperature, 550°C. The vapors expand into vertical 12-inch tubes 12 feet high, speed being reduced to a nominal rate.

269. Hayes, S. D. On the destructive distillation of light petroleum naphthas at low temperatures. Am. Jour. Sci., Ser. 3, vol. 2, 1871, pp. 184–188. A heavy

hydrocarbon oil was obtained from naphtha at a temperature of 212° F. (100° C.) and a pressure of about 50 pounds to the inch. Large iron boilers were used for distilling.

270. Hirsch, Joseph. On the distillation of hydrocarbons. Chem. News, vol. 18, July 31, 1868, pp. 51–52. Discusses "cracking." In distillation of large quantities of oil (40,000 gallons in a still) cracking takes place at various increasing temperatures below the boiling point of the oil.

271. Krämer, G. Der craking Prozess bei der Verarbeitung rumänischen Erdöls. Petroleum, Jahrg. 3, June 3, 1907–1908, pp. 894–895. Evolution of gas at about 270° in cracking heavy Roumanian oil is due to presence of fatty acids which split off carbon dioxide and furnish high molecular paraffins, which in their turn split up, forming methane and olefins. Author recommends that ordinary distillation be followed up to lighting oils, and that higher boiling constituents be distilled under diminished pressure.

272. Krämer, G., and Spilker, A. Ueber die Zersetzung viscöser Körper (Schmieröle) durch Destillation unter Druck. Ber. Deut. chem. Gesell., Jahrg. 33, Bd. 2, 1900, pp. 2265–2270. Treats of chemical products formed by distillation under pressure. Bakunin when distilled at 450° under 20 atmospheres pressure gave 320 grams of liquid products for 500 grams of raw material. 90 per cent of liquid products distilled over by 280°. Distillate contained large quantities of olefins, also naphthenes and paraffins.

273. Letny, A. Ueber die Einwirkung hoher Temperatur auf Petroleum, Braunkohlentheer und andere ähnliche stoffe. Dingl. poly. Jour. Bd. 229, 1878, pp. 353–365. Light oils were obtained by conducting heavy petroleum through a tube heated to not over 300°. Tar obtained contained many aromatic hydrocarbons.

274. Lewes, V. B. The chemistry of the "cracking" of heavy oils. Petroleum World, vol. 12, 1915, pp. 141–145; Jour. Gas Lighting, vol. 129, 1915, p. 455, 599. Summarizes history of cracking process; discusses chemical products formed by cracking; describes the Burton and the Hall process.

275. ——— Motor fuels. Am. Gas Light. Jour., vol. 103, Sept. 13, 20, 1915, pp. 165–167, 170–171, 178–181. Discusses fuel situation in England and on Continent; gasoline composition; tests for quality; gasoline substitutes. Last paper refers to "cracked" spirits, and presents a good general discussion of history of petroleum cracking.

276. Lissenko, K. Zersetzung des Erdöles beim Erhitzen. Dingl. poly. Jour., Bd. 266, 1887, p. 226. Decomposition goes best at about 400° with slowest possible heating. High-boiling constituents are decomposed into those of low boiling point. The best yield of kerosene is obtained from petroleum residues at 434° to 501°.

277. McAfee, A. M. The improvement of high-boiling pétroleum oils, and the manufacture of gasoline as a by-product therefrom, by the action of aluminum chloride. Jour. Ind. Eng. Chem., vol. 7, September, 1915, pp. 737–741; Met. and Chem. Eng., vol. 13, Sept. 15, 1915, pp. 592–597. By proper control of vapors leaving distilling system and entering condenser, and with time enough for action of aluminum chloride, high-boiling oils can be broken down completely into lower boiling oils. The low-boiling oils are sweet smelling, water white, and composed of saturated compounds. Complete report on McAfee's process and results of many tests of various crude oils.

278. ——— Manufacture of gasoline from low-grade oils. Nat. Petroleum News, vol. 7, April, 1915, pp. 20–24. From 1 to 5 per cent of aluminum chloride is placed in the still with the oil. No pressure used. Large yields of gasoline are obtained from crude oils.

279. **Müller, E.** Ueber pyrogene Zersetzung von Gasöl, Phenol, und Kreosot. Jour. prakt. Chemie, Bd. 166, Aug. 31, 1898, pp. 1–50. Contains little on decomposition of petroleum.

280. **Peckham, S. F.** On the distillation of dense hydrocarbons at high temperatures, technically termed "cracking." Am. Jour. Sci., Ser. 2, vol. 47, January, 1869, pp. 9–16. A criticism of Hirsch's article in Chemical News.

281. ——— Production, technology, and uses of petroleum and its products—Cracking. Bureau of the Census. Tenth Census, vol. 4, 1884, pp. 178–180. Reviews early progress of cracking distillation methods.

282. **Perkin, F. M.** Oil cracking. Chem. Trade Jour., vol. 54, 1914, pp. 63–64. A review.

283. **Rittman, W. F.** The theoretical problems involved in the cracking of oils. Jour. Soc. Chem. Ind., vol. 33, June 30, 1914, pp. 626–628; Chem. Abs., vol. 8, Oct. 10, 1914, p. 3360. Discusses theory of equilibria involved.

284. **Robinson, F. C.** The manufacture of petroleum products. Met. and Chem. Eng., vol. 11, July, 1913, pp. 389–394. Diagram of cracking distillation of crude oil on p. 391. With slow distillation cracking temperature goes up to 675° or 700° F. (357° or 371° C.). Details are given of methods of manufacture of various products, with illustrations.

285. **Schuchow, W., and Gawrylow, S.** Vorrichtungen zur ununterbrochenen fractionirten Destillation von Rohöl und ähnlichen Flussigkeiten, wie auch zur Erzeugung von Gas aus Erdöl und seinen Derivaten. Ztschr. angew. Chem., Jahrg. 6, Apr. 15, 1893, pp. 230–231. Petroleum is driven under high pressure through a worm inclosed in a furnace. The products are distilled. Whether process is simple distillation or decomposition depends on size of tubes of the worm and the speed at which the liquid passes through.

286. **Semenow, —.** Ueber Zersetzungspetroleum. Chem. Repertorium, suppl. to Chem. Ztg., Jahrg. 17, Oct. 7, 1893, p. 265. Description of Ragosin's distillation apparatus. From 20 to 25 per cent of petroleum oil, specific gravity 0.816, is obtained from petroleum residues with this apparatus. Comparison of cracking and ordinary methods of distillation.

287. **Silliman, B.** On naphtha and illuminating oil from heavy California tar. Chem. News, vol. 7, 1868, pp. 171–172. Discusses experiments in cracking California oil.

288. ——— Report on the rock oil or petroleum from Venango County, Pa. New Haven, 1855. Author believed that several of the products of distillation were produced by heat and chemical change during distillation. Report was made several years before "cracking" was recognized.

289. **Singer, Leopold.** Ueber einige Versuche die Crakdestillation betreffend. Chem. Rev. Fett-Harz-Ind., Jahrg. 10, 1903, pp. 271–274; Jahrg. 11, 1904, pp. 3–5. A technical investigation of cracking distillation of Roumanian petroleum. Cracking goes on at temperatures of 200° to 300° and moderate temperatures should be used to obtain best results.

290. **Snelling, W. O.** Gasoline from "synthetic" crude oil. Bull. Am. Inst. Min. Eng., No. 100, April, 1915, pp. 695–704. Crude oil is made from natural hydrocarbons by heating in a still about one-fourth full. The oil is heated to a pressure of 800 pounds, then cooled. Gasoline can be removed from product by distillation.

291. **Thorpe, T. E., and Young, John.** On the combined action of heat and pressure upon paraffins. Proc. Roy. Soc., London, vol. 20, 1871–1872, pp. 488–491; vol. 21, 1872–1873, pp. 184–201. Decomposition of paraffins to olefins by heat and pressure. Decomposition did not succeed without distillation.

292. ——— Preliminary notice on the production of olefins from paraffin by distillation under pressure. Proc. Roy. Soc. London, vol. 19, 1870–1871, pp. 370-

371; Chem. News, vol. 23, Mar. 17, 1871, p. 124. Short note. Paraffin exposed to a high temperature in a closed vessel is almost completely resolved, with evolution of little gas, into hydrocarbons that remain liquid at ordinary temperatures.

293. Thorpe, T. E., and Young, John. Ueber die vereinigte Wirkung von Wärme und Druck auf die Paraffine. Ber. Deut. chem. Gesell., Jahrg. 5, Bd. 2, 1872, pp. 556–559; Liebig's Annalen, Bd. 165, 1873, pp. 1–28.

294. Vohl, H. Die fractionirte Destillation bei constantem Niveau behufs der Trennung der in den Erd-und Theerölen enthaltenen Kohlenwasserstoffe zur Erzielung einer grösseren Brennöl-Ausbete. Dingl. poly. Jour. Bd. 177, 1865, pp. 133–139.

295. ——— Die trockene Destillation bituminöser Fossilien behufs Darstellung fester und flüssiger Kohlenwasserstoffe, welche als Beleuchtungs Materialien sowohl, wie zu Maschinen Schmiere und Darstellung von Farben benutz werden. Dingl. poly. Jour., Bd. 173, 1864, pp. 459–472. Describes generally the distillation of bituminous substances. High temperature caused a rearrangement of atoms, resulting in much simpler compounds.

296. Young, James. Verfahren zur theilweisen Umwandlung der aus bituminöser Kohle gewonnenen Schweröle in Leichtöle. Dingl. poly. Jour., Bd. 183, 1867, pp. 151–152. Heavy oils were changed in part to light oils by distillation under 2 atmospheres pressure.

297. Zaloziecki, R. Beiträge zur Kenntniss des chemischen Verhaltens der Erdöle. Ztschr. angew. Chem., Jahrg. 10, Sept. 15, Oct. 1, 1897, pp. 587–592, 619–623. Two parts: 1. Action of sulphuric acid on petroleum. 2. Action of heat and pressure on petroleum. Petroleums heated in a closed tube to not over 250° undergo intermolecular changes, isomerization, etc., of the hydrocarbons not belonging to the paraffin group.

298. Annonymous. The Burton process of cracking oils. Pet. World, vol. 12, 1914, pp. 138, 139. Good description and diagram of apparatus. Pressure of 4 and 5 atmospheres and temperature of 750° to 800° maintained.

299. ——— The "cracking" of oils and its commercial use. Nature, vol. 95, 1915, pp. 231–233. Distinction between decomposition and dissociation. Temperature of dissociation is above boiling point of the oil, and if no pressure is employed oil vaporizes and alteration is small. With residual oils pressure should be regulated to prevent formation of gas.

300. ——— Gasoline distillation from heavy constituents of crude oil. Oil Paint Drug Reporter, vol. 84, Oct. 20, 1913, p. 32G. Good description of Burton process.

PATENTS ON THE CRACKING DISTILLATION OF OIL.

301. Adams, J. H. Oil-converting process. U. S. patent 976,975, Nov. 29, 1910, Jour. Soc. Chem. Ind., vol. 30, Feb. 28, 1911, p. 202. Kerosene and similar oils are cracked by bringing them in contact with an incandescent electric heater. The oil is heated as far as possible by direct contact.

302. Alexejew, G. Destillations Verfahren für Mineralöle vermittelst leichter, bei mittlerer Temperatur flüchtiger Kohlenwasserstoffe. German patent 39,949, Klasse 23, Sept. 23, 1886; Ber. Deut. chem. Gesell., Jahrg. 20, Bd. 4, 1887, p. 609. Light hydrocarbons remaining in vapor form in the cylinder are pumped back through the distillation flask.

303. Bacon, R. F., Brooks, B. T., and Clark, C. W. Making gasoline from petroleum oils boiling at a temperature above 250° C. U. S. patent 1,131,309, Mar. 9, 1915. The oil is cracked at 350° to 500° C. under pressures of 4 to 20 atmospheres. Heating surface for cracking is about one square inch

for each 4.7 cubic inches of oil under treatment. Oil is heated in an upright iron pipe, from which tar and coke fall by gravity into a receiving vessel at the bottom.

304. BACON, R. F., AND CLARK, C. W. Treatment of petroleum hydrocarbons. U. S. patent 1,101,482, June 23, 1914. Method of producing hydrocarbons having boiling points of 30° to 150° C. from petroleum. Pressure, 100 to 300 pounds per square inch. Minimum yield 18 per cent of gasoline hydrocarbons.

305. BARBET, E. A. Continuous apparatus for cracking heavy petroleum. French patents 380,282, Oct. 3, 1906; 390,558, July 31, 1907; Jour. Soc. Chem. Ind., vol. 27, Jan. 15, Nov. 30, 1908, pp. 16, 1105. Describes tubes for cracking oil by intense heat.

306. BENTON, G. L. Process of refining crude petroleum. U. S. patents 342,564, 342,565, May 25, 1886. Oil heated to a temperature of 700° to 1,000° F. 371° to 537° C.) under a pressure of 500 pounds per square inch; then caused to expand into a chamber at same temperature.

307. BURTON, W. M. Manufacture of gasoline. U. S. patent 1,049,667, Jan. 7, 1913. Canadian patent 155,567, 1914. Method of cracking petroleum distillates of boiling point 500° F. (260° C.) at temperatures of 650° to 850° F. (343° to 454° C.) and pressures of 4 to 5 atmospheres.

308. ——— Making gasoline from petroleum hydrocarbons boiling above 260° C. U. S. patent 1,105,961, Aug. 4, 1914. Volatile constituents of liquid are distilled at 650° to 850° F. (340° to 450° C.) and vapors are condensed under a pressure of 4 to 5 atmospheres. Volatile constituents of residue are distilled under atmospheric pressure, and distillate thus obtained is distilled under pressure.

309. CLARK, E. M. Method of distilling petroleum. U. S. patent 1,119,496, Dec. 1, 1914. Method of circulating the liquid rapidly in a small stream under pressure of 3 to 7 atmospheres and at a temperature of 650° to 850° F. (343° to 454° C.); resulting vapors taken off and condensed.

310. ———. Treating petroleum distillates boiling at 500° F. (260° C.) or higher to produce hydrocarbons of lower boiling point. U. S. patent 1,132,163, Mar. 16, 1915. The oil is cracked and distilled in a number of stills so operated that pressure in each still is maintained by incondensable gases produced by previous cracking and distillation in another still.

311. COWPER-COLES, S. O. Improved process for the production of petroleum compounds with low flashing points. English patent 27,945, 1908. Paraffin is heated in a still; vapor led through a series of small metal tubes at high temperature (1,700° F.) (926° C.) and condensed.

312. DANCKWARDT, P. Process for the production of gasolene and naphtha from crude oils, petroleum products, tar oil, or similar products. U. S. patent 1,141,529, June 1, 1915. Heated liquid is sprayed into a retort, then withdrawn and passed through a heater, and back to retort. A spray of molten lead, alloy, or fused sodium nitrate or sodium hydroxide is passed into retort.

313. DEWAR, JAMES, AND REDWOOD, BOVERTON. Process for distilling mineral oils and like products. U. S. patents 419,931, Jan. 21; 426,173, Apr. 22, 1890; English patent 10,277, June 24, 1889. Method of distilling petroleum under pressure. Space not occupied by liquid is filled with a gas that can not act chemically on matter treated.

314. ——— Verfahren und Appavat zum destilliren vom Mineralölen und ähnlichen Stoffen. German patent 53,552, Klasse 23, 1889. Chem. Ztg., Jahrg. 14, Nov. 19, 1890, p. 1575. An apparatus for distilling oil under pressure. Air or carbon dioxide is forced into the apparatus.

315. DUBBS, J. A. Dehydrogenating and distilling petroleum or other oils. U. S. patent 1,100,717, June 23, 1914. Air is passed through oil at a temperature at which oxygen of air will combine with hydrogen of hydrocarbons.

316. Dubbs, J. A. Treatment of petroleum. U. S. patent 1,135,506, Apr. 13, 1915. Petroleum is mixed with water under pressure, the mixture vaporized and discharged into a heated chamber in the form of a fine spray. Temperature 300 to 600° F. (148° to 315° C.). Pressure 50 to 600 pounds.

317. Fenchelle, H. E., and Perkin, F. M. Conversion of heavy hydrocarbons into lighter hydrocarbons. English patent 6,547, Mar. 14, 1914. Chem. Abs., vol. 9, 1915, p. 2451. The heavy hydrocarbons are cracked by forcing them under pressure through one or more long, narrow, heated conduits, resulting liquid being cooled under pressure, and allowed to escape at lower pressure into a chamber surmounted by a rectifying column. Temperature, 500 to 600°; pressure, 50 to 60 atmospheres.

318. Gray, J. L. Process of distilling and cracking hydrocarbon oil to increase the yield of gasoline. Canadian patent 153,966, Feb. 9, 1914. The oil is passed through a bath of molten metal at a temperature sufficient to cause cracking; resulting vapors are condensed.

319. Greenstreet, C. J. Process of treating heavy hydrocarbon oils. U. S. patents 1110923, 1110924, Sept. 15, 1914.

320. ——— Process of manufacturing olefins and their oxidation products. U. S. patent 1110925, Sept. 15, 1914; English patent 16,452, July 13, 1912. These patents cover method of forcing crude oil mixed with steam through a long coil of pipe at cherry-red heat.

321. Hall, W. A. Obtaining motor spirits by cracking heavy hydrocarbon oils. English patent 2,948, Feb. 4, 1914; Chem. Abs., vol. 9, July 20, 1915, p. 1994. Oil is passed through a coil at 600°, at such a rate that only about 25 per cent is converted into gas, and superheating is avoided. The product, consisting mainly of unsaturated hydrocarbons, is passed through alcohol, and resulting mixture is condensed.

322. Humphreys, R. E. Method of distilling hydrocarbons. U. S. patent 1,119,700, Dec. 1, 1914. Hydrocarbon oils boiling over 500° F. (260° C.) are distilled under a pressure of over 4 atmospheres; heavier fractions are condensed and returned to still for further treatment.

323. Hyndman, H. M. Cracking petroleum oils. French patent 462,484, Sept. 11, 1913. The oils are passed through a heated spiral at such a temperature that heavy constituents are converted into lighter hydrocarbons. The temperature is described as "a cherry-red heat," and pressure is used.

324. Kittle, H. A. Improvements in or relating to the vaporization of oil and other volatile substances. English patent 8,336, Apr. 6, 1909. Oil is vaporized at a temperature above boiling point of highest boiling constituent. Velocity of flow through apparatus is not less than velocity at which same oil would spread if brought into contact with hot vaporizing surface. The oil may be heated in a bath of molten metal.

325. Krey patent. *See* Riebecksche Montanwerke.

326. Laing, John. Improvements in the apparatus for the destructive distillation of mineral oils. English patents 4,120, Mar. 17, 11,079, July 16, 11,757, July 28, 1890; Jour. Soc. Chem. Ind., vol. 10, June 30, July 31, 1891, pp. 534, 631. Gases or vapors from the oil-still are passed through a superheater, and further split up. A lighter distillate is thus produced than if the gases or vapors had gone directly to the condenser.

327. ——— Improvements in apparatus for the destructive distillation of mineral oils and the like. English patent 23,446, Oct. 24, 1911; German patent 260,858 Klasse 23 b, 1911. A still is divided into compartments with tubular connections for leading vapors and oil from compartment to compartment, and condensers fitted in connections. A water trap is interposed between each condenser and its succeeding compartment. Connections for leading back uncondensed gases to be reabsorbed by vapors coming from the still.

328. Leffer, L. G. Process for transforming high boiling hydrocarbons into hydrocarbons of lower boiling point. English patents 2,328, Jan. 29; 19,051, Aug. 20, 1912; 4,140, Feb. 18, 1913; French patents 439,476, Jan. 29, 1912; 454,580, 1913; Jour. Soc. Chem., Ind., vol. 31, Aug. 15, 1912, p. 711; vol. 32, Mar. 15, Sept. 15, Sept. 30, Dec. 15, 1913; pp. 224, 861, 903, 1098. Petroleum is distilled at high pressure in an atmosphere of an inert gas at temperature not over 410° C. The hydrocarbons also may be carried into fractionating chamber as spray or mist by a current of inert gas at a temperature of about 400° C. (*See* English patent 4,140, Feb. 18, 1913.)

329. Parker, J. H. Process for the treatment of oils. U.S. patent 958,820, May 24, 1910. Petroleum in finely divided condition is forced under pressure through a series of chambers, heat sufficient to generate vapor under pressure being imparted to chambers. Petroleum escapes from each chamber to succeeding chamber under a definite difference in pressure. Light oils are formed.

330. Ragosin, V. I. Distilling, fractioning, and cracking mineral oils, and obtaining various products therefrom; a method of, and apparatus for that purpose. English patent 1,411, Jan. 18, 1898; Jour. Soc. Chem. Ind., vol. 17, May 31, 1898, p. 445. Oil is mixed with benzene, and run into a cracking chamber.

331. Renard, Georges. Process and apparatus for the transformation of petroleum and similar hydrocarbons into products having a lower boiling point. English patent 3,413, Feb. 10, 1913; French patent 462,286, Sept. 8, 1913; Jour. Soc. Chem. Ind., vol. 33, Apr. 15, Apr. 30, 1914, pp. 345, 412. The liquid oil is heated in coiled pipes under a pressure of 40 to 50 atmospheres at a temperature of 400° to 450°. "The hydrocarbon remains in a liquid state throughout, but is broken down into fractions of lower boiling point."

332. Riebeckshce Montanwerke. Verfahren zur Abscheidung von leichten Kohlenwasserstoffen und zur Gewinnung dickflüssiger oder asphaltartiger Oele aus paraffin Oelen etc. durch Destillation unter höherem Druck. German patent 37,728, Klasse 23, 1886. Chem. Ztg., Jahrg. 10, Dec. 29, 1886, p. 1622; Jour. Soc. Chem. Ind., vol. 6, Apr. 29, 1887, p. 282. The oils are distilled in cast-steel retorts under a pressure of 3 to 6 atmospheres. A large proportion of light oils is formed, and residue can be used for lubricating or asphalt making.

333. Seigle, Adolphe. Apparatus for treating hydrocarbons. U. S. patents 567,751, 567,752, Sept. 15, 1896. A series of vaporizing chambers and a series of superheating chambers containing inert material, so arranged that the hydrocarbons are successively heated in the vaporizing chambers, and passed to the superheating chambers. Both series of chambers are immersed in a bath of molten metal.

334. Turner, C. W. Distilling and cracking oils. U. S. patent 1,046,683, 1912; English patent 25,832, Nov. 11, 1912. Oil with steam is forced by pressure through heated coils, and resulting vapor submitted to a decomposing temperature, the products passing to vessels of larger diameter.

335. Von Groeling, A. E. Improved process of and apparatus for distilling and cracking heavy and light hydrocarbon residues. English patent 10,213, 1913. Hydrocarbon vapors and liquid hydrocarbons are passed together through cracking tubes at temperatures from 350° to 500° C., and under a pressure of 1 to 1½ atmospheres.

336. Washburn, C. H. Producing lighter hydrocarbons from kerosene or heavier hydrocarbons. U. S. patent 1,138,266, May 4, 1915. Petroleum is mixed with 25 to 100 per cent of its weight of water, and heated in a retort to 600° to 950° F. (315° to 570° C.) under a pressure of 3 to 5 atmospheres.

337. Young, James. Improvements in treating hydrocarbon oils. English patent 3,345, Dec. 27, 1865. The heavier hydrocarbon oils are distilled in steam boilers under a pressure of 20 pounds to the inch. Oils of lower specific gravity are produced.

338. Zerning, H. Cracking mineral oil and other hydrocarbons for use as fuel for internal combustion engines. English patent 731, Jan. 9, 1913; French patent 452,919, Jan. 6, 1913. The raw materials are treated with gases or vapors obtained by injecting water and hydrocarbons into a pipe heated to 300° to 400° and leading vapors through dark, red-hot pipes into a cooler to condense oil, tarry matter, and water. Natural gas or gases obtained by destructive distillation of organic substances may be used for first treatment.

PATENTS ON THE PRODUCTION OF LIGHT OILS FROM PETROLEUM BY USE OF CATALYZERS.

339. Abel, C. D. Treatment of hydrocarbons for their purification and conversion into other products. English patent 4,769, Dec. 15, 1877. Low-grade petroleum is treated with aluminum chloride, or a mixture of aluminum chloride and metallic oxides, at 100° to 600° C.

340. Bergius, F. Production of light hydrocarbons from heavy hydrocarbons. English patent 4,574, Feb. 21, 1914; French patent 470,551, Apr. 6, 1914. The heavy hydrocarbons are subjected to action of hydrogen, nitrogen, carbon monoxide, or methane at a pressure of 100 atmospheres, while heated to a temperature below 420° C.

341. Breitenlohner, I. I. Die Torföle und ihre Aufbereitung nach einem patentirten Verfahren. Dingl. Poly. Jour., Bd. 167, 1863, pp. 378–390. Oils are cracked by leading their vapors through a red-hot iron tube, or into a chamber filled with red-hot coke, slag, or other material.

342. Continental-Caoutchouc and Gutta-Percha Co. Conversion of higher boiling mineral oils into lower boiling products. English patent 7,112, Mar. 20, 1914; Jour. Soc. Chem. Ind., vol. 34, Aug. 16, 1915, p. 826; Chem. Abs. vol. 9, Sept. 10, 1915, p. 2452. The oils are passed over products obtained from aluminium halides and hydrocarbons, or halogenalkyls as catalyzers. The catalyzers may be absorbed in a porous substance.

343. Day, D. T. The process of refining and purifying hydrocarbon oils. U. S. patent 826,089, July 17, 1906. Hydrogen or absorbable hydrocarbon is added under pressure, in the presence of porous absorptive substances capable of absorbing hydrogen or a hydrogen-carrying gas or vapor. Palladium black or platinum sponge may be used.

344. Ellis, C. Cracking heavy oils. English patent 25,631, Nov. 8, 1912. Unde, Int. Conv. Apr. 1, 1912. The oils are injected into a chamber filled with firebrick, alumina, or other refractory material. Sufficient air is injected through other nozzles to serve for combustion of a small part of this oil, the remainder being vaporized and cracked under a pressure of 30 to 60 pounds per square inch. The refractory material is coated with a layer of catalytic material, such as thorium, vanadium, or precipitated alumina, attached by means of feldspar or other frit.

345. Friedel, C., and Crafts, J. M. Improvements in the treatment of hydrocarbons for their purification and conversion into other products. English patent 4,769, 1877; Chem. Ind., Jahrg. 1, 1878, p. 411. Patent filed by C. D. Abel. The hydrocarbons are treated with aluminium or other metallic chlorides at temperatures of 100° to 600° C. Metallic oxides also may be added. Petroleum is converted into light oil; naphthalene forms benzene, toluene, and other products.

346. Gray, G. W. Converting higher-boiling into lower-boiling petroleum hydrocarbons. English patents 17,838 and 17,839, Aug. 5, 1913. Oil is heated in presence of anhydrous aluminium or ferric chloride. At 325° to 350° F. (162° to 176° C.) with 5 per cent of aluminium chloride, 70 to 80 per cent of napththa was obtained. In second patent a temperature of 400° to 600° F. (204° to 315° C.) was used

347. HALL, W. A. Conversion of heavy into lighter hydrocarbons. English patents 17,121 (July 25), and 24,491 (Oct. 28), 1913; 437, Jan. 7, and 7,282, Mar. 23, 1914. Hydrocarbon vapors are heated to above 600° in presence of a suitable catalyser, and under a pressure of 5 atmospheres. Resulting vapors are passed through an expansion chamber. Nickel, cobalt, silver, platinum, chromium, manganese, or their oxides may be used as catalyzers. Yield is 50 to 70 per cent of oil specific gravity 0.765. Resulting vapors may be purified by passing through fullers' earth, bauxite, etc.

348. HIRSCHBERG, LEON. Improvements in or relating to the treatment of hydrocarbons. English patent 4,573, Feb. 21, 1914. Heavier hydrocarbons are converted into lighter hydrocarbons by contact in a finely divided state with a catalyzer, as chromium oxide, at dull red heat. Pressure, 15 to 55 pounds per square inch.

349. HUMPHREYS, R. E. Obtaining low boiling hydrocarbons from liquid paraffin hydrocarbons of petroleum which boil at 500° F. (260° C.) or higher. U. S. patents 1,122,002; 1,122,003, Dec. 22, 1914. A portion of liquid is distilled under 4 atmospheres, or higher, pressure at a temperature of 340° to 435° C. (650° to 850° F.), in a still containing steel, brass, or copper plates immersed in liquid.

350. LAMPLOUGH, F. Process and apparatus for the conversion of heavy hydrocarbons into lighter hydrocarbons. English patents 19,702, Aug. 28, and 28,101, Dec. 5, 1912; Jour. Soc. Chem. Ind., vol. 32, July 31, Nov. 15, 1913, pp. 743, 1001. A mixture of oil and 10 per cent of water is passed through a series of U-tubes containing metallic nickel rods heated to dull redness. The resulting vapors are condensed and separated.

351. MCAFEE, A. M. Process of improving oils. U. S. patent 1,127,465, Feb. 9, 1915. Oil is distilled in the presence of 1 to 5 per cent of aluminium chloride. Large yields of gasoline are obtained. Temperature, below 600° F. (315° C.); time, 36 to 48 hours.

352. MOELLER, J., AND WOLTERECK, H. C. Production of light oils from crude, heavy, or lighting oils. English patent 16,611, July 19, 1913. Vapors of crude oil are mixed with steam at 700° to 800° C. under atmospheric pressure, and passed through tubes filled with coke heated to 600° to 700° C.

353. NEW OIL REFINING PROCESS, LTD., AND NEILSON, R. G. Improvements in apparatus for converting mineral oil into volatile spirit. English patents 13,726, June 13, 1913; 20,074 and 20,075, Sept. 3, 1912; 28,460, Dec. 18, 1911. Oil and water are decomposed together in the presence of scrap iron, iron carbide, copper, aluminium, bauxite, broken bricks, coke, alumina. Temperature, 1,000° to 1,200° F. (537° to 648° C.).

354. NOAD, JAMES, AND TOWNSEND, E. Improvements in the treatment of hydro carbon oils or the like for the production of volatile spirit. U. S. patent 971,468, Sept. 27, 1910; English patents 13,675, June 27, 1908; 3,607, Feb. 13, 1912. Oil and water are vaporized and decomposed together in the presence of highly heated iron. Temperature, 900° F. (482° C.).

355. PLANES, LTD., AND THOMPSON, W. P. Cracking petroleum and other hydrocarbons. English patents 5,245, Mar. 1, and 21,273, Sept. 20, 1913. The vapors are heated in a cracking still, with a catalyzer, such as finely divided nickel, hydrogen, or purified water gas. The hydrogen or water gas may be passed through the petroleum in the presence of finely divided nickel immersed in the liquid. Temperature, 300°; pressure, 5 to 100 pounds per square inch.

356. PORGES, S., STRAUSKY, S., AND STRACHE, N. Process and apparatus for converting heavy hydrocarbons into light hydrocarbons. English patent 11,420, May 8, 1914; Jour. Soc. Chem. Ind., vol. 34, Aug. 16, 1915, p. 826. A mixture of vaporized oil and steam is passed over a catalyzer (iron oxide or oxide of another metal) at 500° to 600° C.

357. Sabatier, Paul, and Mailhe, A. Converting heavy hydrocarbons into lighter hydrocarbons. U. S. patent 1,124,333, Jan. 12, 1915. Crude oils are passed over finely divided catalytic metals, such as tungsten, tantalum, nickel, or cobalt, heated to redness by an electric current. The light unsaturated hydrocarbons obtained may be hydrogenated by contact with nickel, copper, cobalt, iron, or platinum, heated to 200° to 300° C.

358. ——— Treatment of liquid hydrocarbons for the production of lighter hydrocarbons. English patent 16,791, July 14, 1914; Jour. Soc. Chem. Ind., vol. 34, Aug. 16, 1915, p. 826. Liquid hydrocarbons are converted into low boiling (150° C.) compounds by passing them over heated catalyzers, such as finely divided metals or metallic oxides or refractory substances free from silica. Carbon deposits are removed by a current of steam.

359. Shedlock, J. J., Optime Motor Spirit Synd. (Ltd.), London. Treatment of liquid hydrocarbons for their purification and the production of motor spirit and by-products. English patent 1,878, Jan. 23, 1914; Jour. Soc. Chem. Ind., vol. 34, June 15, 1915, p. 604; Chem. Abs., vol. 9, July 20, 1915, p. 1994. A mixture of oil and an emulsion of oil and water is passed through a vessel containing a catalyzer, such as iron, steel, or nickel, heated to 260° to 480° C., and the resulting oil and gas passed into an expansion chamber.

360. Standard Oil Co. Distilling petroleum residues. English patents 29,862, Dec. 28, 1912; 6,593, Mar. 16, 1914; French patent 452,604, Dec. 28, 1912; Chem. Abs., vol. 9, Sept. 10, 1915, p. 2452. Petroleum residues are distilled at 650° to 850° F. (343° to 454° C.) under a pressure of 4 to 5 atmospheres; residue from this operation is distilled to dryness under atmospheric pressure, and the distillate submitted to a cracking.

361. Testelin, A., and Renard, G. Manufacture of a new spirit by the isomerisation of petroleum. English patent 16,881, Aug. 11, 1908; German patent 268,176, Klasse 23b, 1908; French patent 393,554, Aug. 8, 1908. Jour. Soc. Chem. Ind., vol. 28, Feb. 15, 1909, pp. 133, 695. A thin stream of petroleum is injected by high-pressure, superheated steam into a coil heated to 400° or 450° C. From the coil the vapors pass through a tube over burnt fire clay at full red heat.

362. ——— Apparatus for making volatile hydrocarbons from petroleum. U. S. patent 1,138,260, May 4, 1915; German patent 226,958, Klasse 23b, 1909. Petroleum is heated with steam at high pressure and passed over a refractory material at red heat. Temperature, 400° to 450° C.

363. Valpy, O. H., and Lucas, O. D. Process of cracking oils by bringing a mixture of oil and steam into contact with a heated catalyzer, such as nickel or other metal or metallic oxide. English patents 20,470, Sept. 10, 1913; 5,847, Mar. 7, 1914; 12,653, May 22, 1914; 18,923, 1914.

364. White, C. Process of cracking oils. English patent 5.434, 1914. Mineral oils and residues are brought in a liquid state onto calcium oxide, or calcium oxide containing carbon, at a temperature of 400° to 650°. Pressure is reduced to avoid formation of gas. Steam or water is not added.

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